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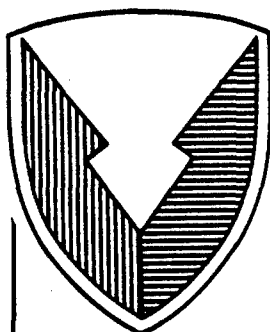
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Technical Report



No. 13496

BY-PASS OIL FILTER TEST TWO-YEAR PROGRAM (U)

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<p>This two-year oil filter test program was conducted to evaluate the effectiveness of by-pass oil filters in maintaining the cleanliness and lubrication qualities of the engine oil, during normal troop training operations. The engine oil characteristics for M939A1 trucks, with by-pass oil filters, were compared with the oil analysis characteristics of the used oil taken from similar M939A1 trucks which had only the standard full-flow oil filters. The engine oil samples which were obtained from the subject trucks at 60-day intervals, were given a laboratory analysis for the following: Wear-metal concentrations for following elements: copper, iron, lead and aluminum, the depletion of certain oil additives, oil viscosity measurements, concentrations of ferromagnetic particles (by average size), and the TAN & TBN values.</p> <p>The results of these laboratory tests showed that by-pass oil filters did not appear to have any adverse effects on the calcium, magnesium, silicon, or zinc additives used in these</p>					
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engine oils. Also, there were no apparent effects on the engine oil viscosities due to the usage of the by-pass oil filters. Overall, the by-pass oil filters did not appear to have any effects on the oils TAN increases or the TBN decreases during the program.

The results of the DR-Ferrograph tests on the engine oils showed that the concentrations of ferromagnetic particles (by average sizes) were slightly less for oil from the trucks with the by-pass oil filters, than for the oil from the 5-ton trucks with the standard full-flow oil filters.

The results of a cost comparison, or cost effectiveness study, for the subject by-pass oil filters, when installed on the subject 5-ton trucks, showed that the potential cost savings would be relatively small. That is to say, that this study resulted in a potential savings of less than one-half ($1/2$) of one engine oil change (in a two-year period), which would be less than \$48.00 per vehicle. This potential savings would be much less than the cost of installation of these by-pass oil filters, which costs were in excess of \$200.00 per vehicle. It must be mentioned here that the subject two-year test program did not provide for any long-term engine wear studies or conclusions. However, based on the test results mentioned above, and the cost comparison studies, it has been concluded that the usage of by-pass oil filters on the subject 5-ton M939A1 trucks would not be cost effective.

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1.0. INTRODUCTION

This technical report has been prepared by the U. S. Army Tank-Automotive Command (TACOM), under Contract Numbers DAAE07-87-L-R006 and R007, with technical support from the U. S. Army Belvoir Research, Development & Engineering Center (STRBE-VF), and the Belvoir Fuels & Lubricants Research Facility (BFLRF), located at San Antonio, TX, under Contract No. DAAK70-87-C-0043. This two-year oil filter test program was designed to evaluate the effectiveness of by-pass oil filters from two companies (Filter "A" and Filter "B"), in maintaining the cleanliness and lubrication qualities of the engine oil during normal operating conditions. These by-pass oil filters were added to the standard full-flow oil lubrication systems used on the NHC-250 engines in the M939A1 cargo trucks.

These tests were conducted on a fleet of new M939A1 trucks which were employed by the 9th Infantry Division for troop training at Fort Lewis, WA. The analysis method used for these engine oil samples, taken from the M939A1 trucks, was to conduct oil analysis at the Fort Lewis Army Oil Analysis Program (AOAP) laboratory, along with analysis of duplicate samples being sent to the BFLRF laboratories. Comparisons were then made between the oil analysis data obtained from engines with the "by-pass" oil filters added, and the data obtained from engines with the standard full-flow oil filter systems.

2.0. OBJECTIVES

The primary goal of the two-year oil filter program was to determine the effectiveness of the added "by-pass" oil filters in maintaining the lubricating qualities and the overall cleanliness of the engine oils, under normal engine operating conditions. These oil characteristics, for trucks with by-pass oil filters, were compared with the oil analysis characteristics of used oil taken from similar trucks having only standard full-flow oil filters.

3.0. CONCLUSIONS

After the completion of the two-year oil filter test program, the following evaluations were made:

a. Used Oil Wear-Metal Concentrations: The M939A1 trucks with the added by-pass oil filters installed exhibited a slight reduction in wear-metal concentrations (over a two-year period), than the standard M939A1 trucks which had only the standard full-flow oil filters. However, these differences were considered to be due primarily to the 14.8 percent oil dilution factor for the by-pass oil filters. (See following para.).

b. Oil Dilution Factor There were many differences seen in the wear-metal concentrations for the trucks with by-pass oil filters, as compared to the results for trucks with the standard full-flow oil filters. These results showed generally lower concentrations for trucks equipped with by-pass oil filters (See Figs. 3-1 & 3-2). However, this was considered to be due primarily to the fact that the M939A1 trucks with the by-pass oil filters required an additional 4 qts. of 15W40 oil in the engine oil system (which has a std capacity of 27 qts). Therefore, this represents an oil dilution factor of 4 qts in 27 qts, or approximately 14.8 percent.

c. Changes & Depletions in Oil Additives In summary, the by-pass oil filters did not appear to have any adverse effects on the calcium, magnesium, silicon, or zinc additives in the truck engine oils.

d. Oil Viscosity Measurements There were no apparent effects on the engine oil viscosities due to the usage of the by-pass oil filters. In addition, there were only negligible instances, or occurrences, of fuel dilution in these engine oils during the two-year test program.

e. TAN & TBN Values Overall, the by-pass oil filters did not appear to have any effects on the oil's Total Acid Number (TAN) increases or on the Total Base Number (TBN) decreases, during the test program.

f. DR - Ferrograph Tests The test results showed that the WPC values (concentrations of ferromagnetic particles by average size) were less for the used oils from trucks with the by-pass oil filters than the used oils from the 5-ton trucks with standard full-flow oil filters. (See Table 3-1, & see Appendix A).

4.0. RECOMMENDATIONS

4.1 Based on the installation cost figures for the by-pass oil filters and the potential savings of less than one-half (1/2) of one (1) oil change during a two-year test period, the usage of these filters would not be cost effective.

4.2 Monitoring of the TAN and TBN values for used oils is considered to be an essential part of proper used-oil analysis. Therefore, it is recommended that TAN and TBN oil analysis should be incorporated as a part of the normal AOAP laboratory analysis procedures.

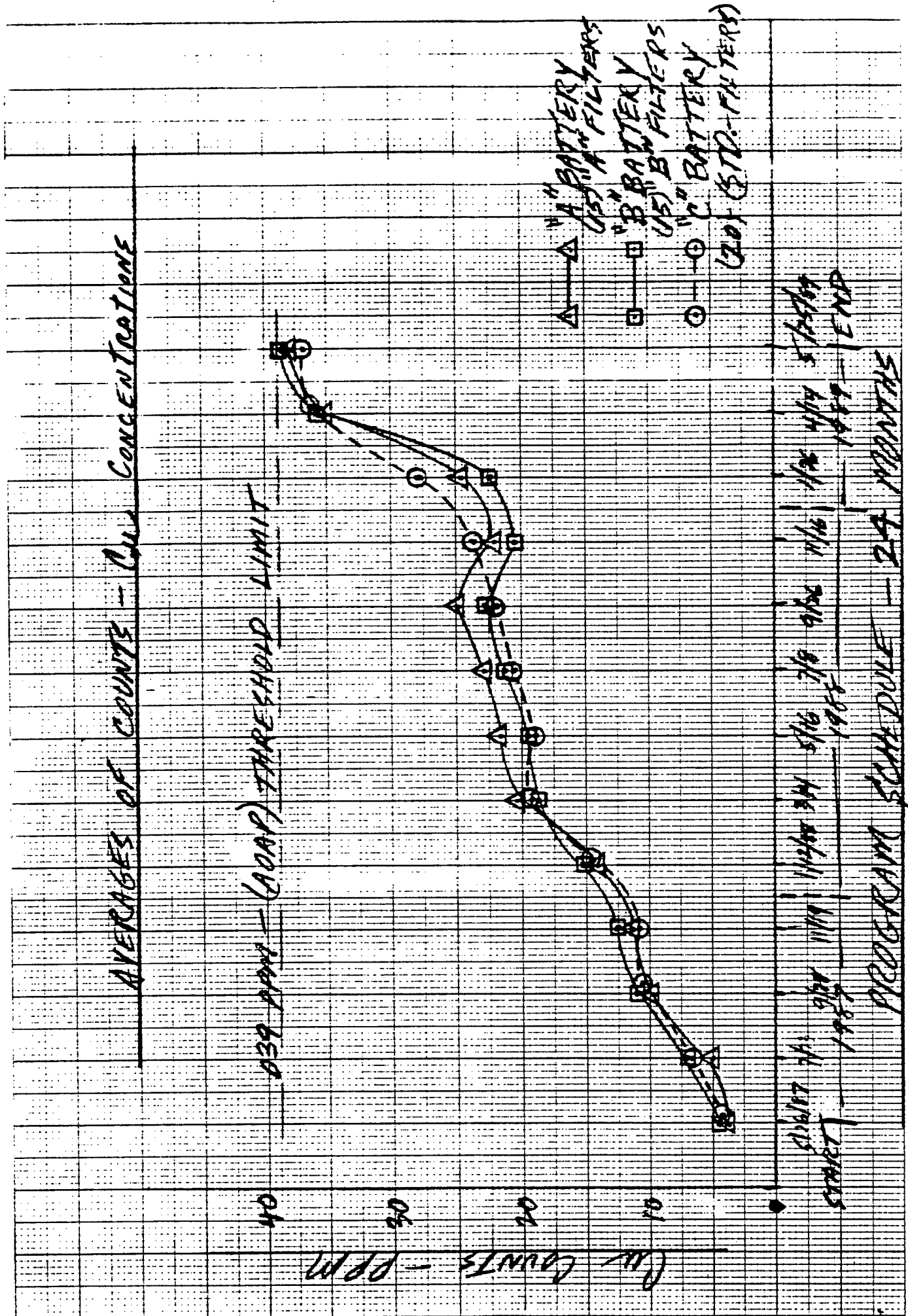


FIGURE 3-1. Averages of Counts
Cu - Concentrations

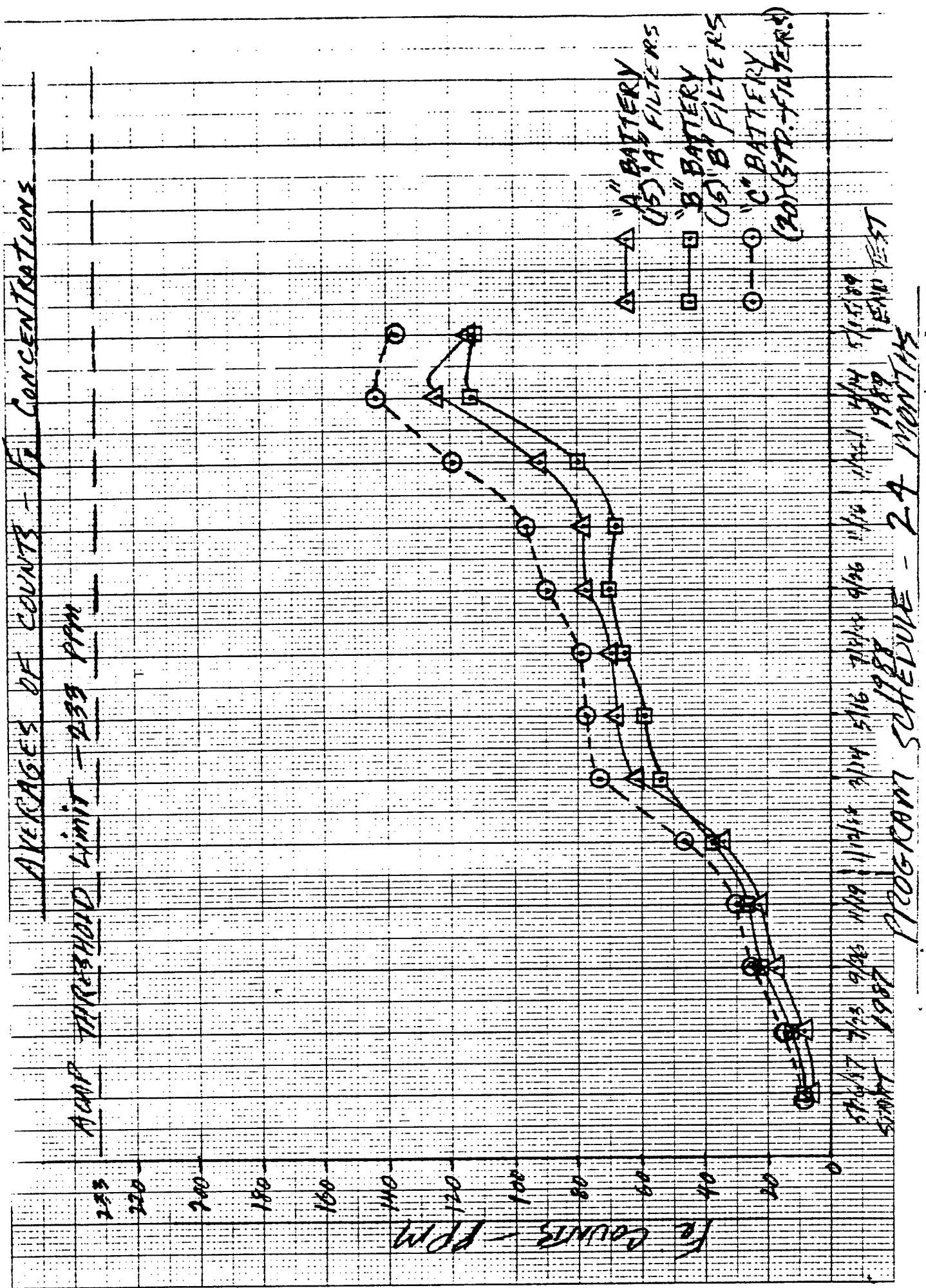


FIGURE 3-2. Averages of Counts
Fe - Concentrations

FIGURE 3-2

TABLE 3-1. DR - FERROGRAPH TEST RESULTS

TABLE 3-1

DR - FERROGRAPH TEST RESULTS

(FLEET AVERAGING VALUES)

VEHICLE DESCRIPTION (+) FILTER TYPE	OIL SAMPLES - AND DATES OBTAINED					
	1/12/88 #5	3/14/88 #6	5/16/88 #7	7/8/88 #8	11/16/88 #10	1/26/89 #11
"A" - BATTERY VEH'S W/ "A" FILTERS (WPC) =	D _L 12.9 D _S 7.5 20.4	D _L 16.2 D _S 10.7 26.9	D _L 14.6 D _S 10.6 25.2	D _L 12.5 D _S 10.8 23.3	D _L 12.4 D _S 9.2 21.6	D _L 12.3 D _S 9.3 21.6
	D _L 29.7 D _S 11.9 41.6					
"B" - BATTERY VEH'S W/ "B" FILTERS (WPC) =	D _L 18.6 D _S 10.5 29.1	D _L 14.4 D _S 10.7 25.1	D _L 13.1 D _S 9.8 22.9	D _L 13.2 D _S 10.2 23.4	D _L 10.5 D _S 7.2 17.7	D _L 15.4 D _S 10.9 26.3
	D _L 15.6 D _S 9.0 24.6					
"C" - BATTERY VEH'S W/STD. FULL-FLOW FILTERS (WPC) =	D _L 22.0 D _S 14.3 36.3	D _L 28.4 D _S 15.9 44.3	D _L 23.8 D _S 16.2 40.0	D _L 20.4 D _S 12.8 33.2	D _L 33.0 D _S 19.6 52.6	D _L 35.8 D _S 14.3 50.1
	D _L 27.2 D _S 19.7 46.9					

LEGEND

(WPC) - Wear Particle Concentration

D_L - Concentration of the larger ferromagnetic particles (5 u m and larger).D_S - Concentration of the smaller ferromagnetic particles (smaller than 5 u m).

5.0. DISCUSSION

5.1. BACKGROUND

5.1.1. USED OIL WEAR-METAL CONCENTRATIONS

During the first 12 months of the oil filter test program, it was noted that the basic concentration in parts per million (ppm) of the three metals of copper (Cu), iron (Fe) and lead (Pb), were quite similar for all three groups of test vehicles, that is the "A," "B" and "C" battery trucks. Also, there were no significant differences shown between the Wear Metal Concentrations (WMC) on the trucks with the by-pass oil filters, "A," "B" and "C" battery trucks, with only the standard full-flow filters.

5.1.2. OIL-DILUTION FACTOR

There is an oil-dilution factor which must be considered when any comparisons are made between full-flow and by-pass oil filter data presented in this report. This is because the installation of the added by-pass oil filters on the NHC-250 engines added approximately 4 qts of SAE 15W-40 oil to each engine. This additional oil was required to completely fill up the by-pass filter housings and elements. The actual capacities of these by-pass oil filters were as follows:

Filter "A" capacity is approx. 2.75 qts.

Filter "B" capacity is approx. 3.25 qts.

However, despite these slight differences in capacity, a decision was made, prior to the start of the test program, to add a full four (4) qts. of SAE 15W-40 oil to each truck engine with a by-pass oil filter installed, for purposes of oil quantity uniformity. The standard oil lubrication system capacity for the NHC-250 engine, with its standard full-flow oil filter, is established at 27 qts. Therefore, as can be seen by a simple ratio of 4 qts added to the basic 27 qts., this represents an oil-dilution factor of approximately 14.8 percent. And, as a result of this, it becomes necessary to employ this dilution factor, when an accurate comparison of the relative concentrations of wear metals is desired.

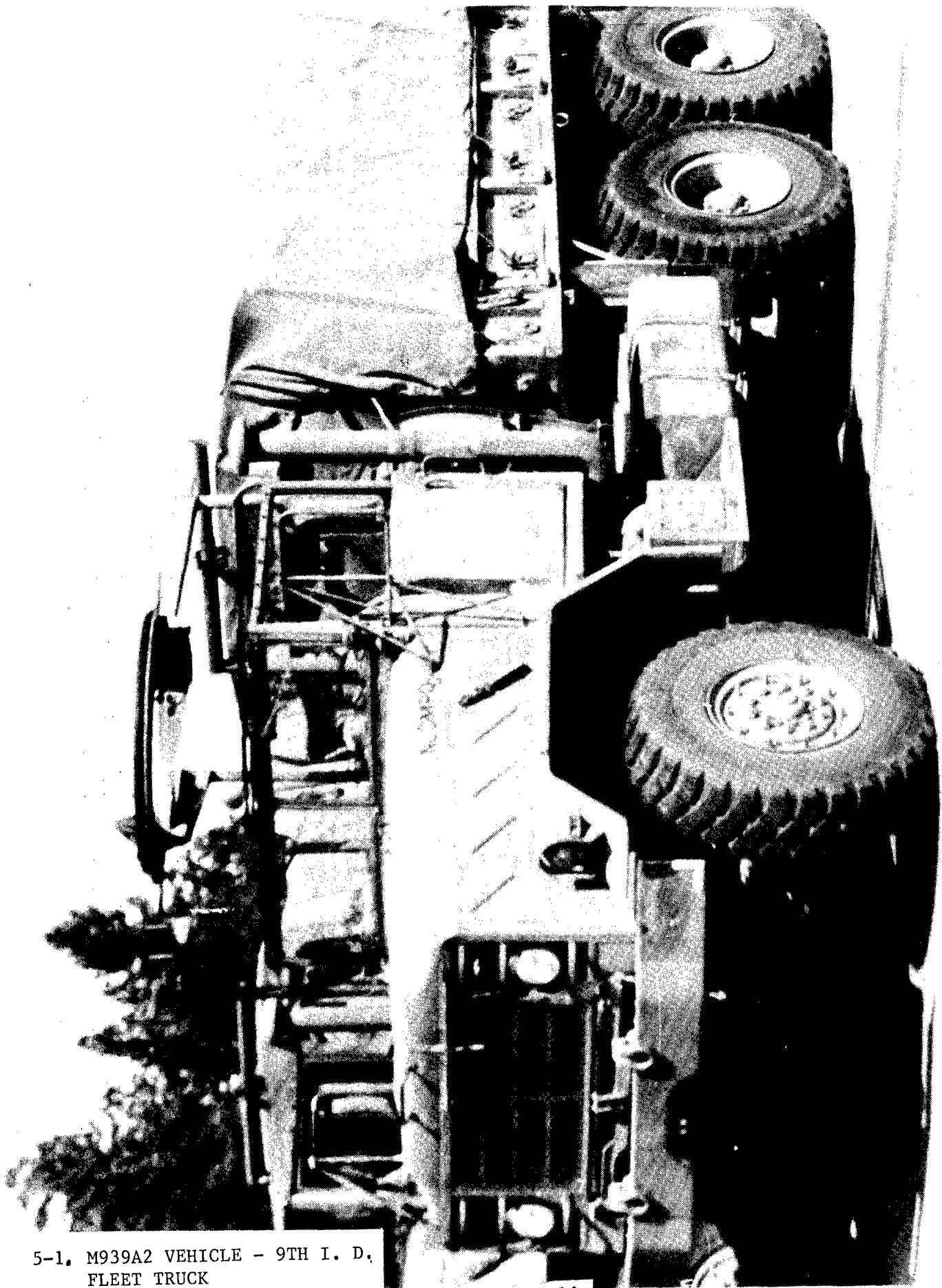
The test data in this report is shown in the "as monitored," or "as received" condition, with the 14.8 percent oil dilution factor taken into consideration by means of specific examples, as are indicated for the various data comparisons which are made in this report.

In general, the increases in WMC were not excessive in any of the 50 trucks in this test program, and the WMC did not exceed the "AOAP" threshold limits. It was noted in some of these trucks that the WMC levels for the copper (Cu) element were approaching the "AOAP" threshold limit of 039 ppm. This condition would normally indicate a need for oil change in those engines; however, as these Cu threshold limits are set to occur at a relatively low concentration, there was no indication of potential engine problems at the 12-month point in the test.

This data and conclusions were determined by the fact that all 50 vehicles in this program used MIL-L-2104D engine oil (SAE 15W-40) that had been installed initially in these trucks at the beginning of the test program (May 1987).

As the test program continued with these vehicles for the second year (12-month period), it was noted that the WMC levels for the Cu and Fe elements were gradually increasing to higher levels, and in the case of the Cu WMC to the AOAP threshold limits. As can be seen in Figure 3-1, the Cu WMC in ppm had increased in a number of the M939A1 vehicles (See Fig. 5-1.) up to a ppm count which met or exceeded the AOAP threshold limit of 039 ppm. In a significant number of these trucks, the engine oil exhibited Cu concentrations ranging from 035 to 040 ppm. It is interesting to note that by the completion of the two-year program, there were a total of 30 vehicles which required engine oil change, primarily due to the high Cu concentrations. Also, it is worthy to note that in the subject NHC-250 engines, the basic wear metals are caused by wear occurring in the engine main bearings, piston pin bushings and from the camshaft bushings. However, it should be mentioned here, that it is possible to acquire increases in the Cu concentration due a Cu additive used in some engine oils by certain oil formulator companies, as an anti-oxidant agent. This problem of a high Cu concentration in new oils can be determined, of course, by taking "AOAP" lab samples of new source oil, prior to use in truck engines. (See Fig. 3-1 & Fig. 3-2).

In regard to the above statement on the Cu concentration, which can be present in new source oils, it should be noted here that the source oils which were used for the subject M939A1 trucks were very low in Cu concentrations (See Table 5-4). Therefore, this factor of Cu concentrations showing up due to Cu additives in source oils was not a problem during the subject test. However, there was a single exception (to Cu additives) which showed up in a final source-oil sample (taken in May 1989). This one source sample No. AL-18739 (see Table 5-4) exhibited a Cu concentration of 054 ppm (which is high). This source oil, however, was used only for engine oil changes which took place in May 1989, at the end of test. Therefore, these oil changes did not affect the "AOAP" oil sampling data which was obtained for the subject two-year program.



5-1. M939A2 VEHICLE - 9TH I. D.
FLEET TRUCK

5.1.3 The "AOAP" specified threshold limit of 039 ppm for Cu is considered to be somewhat low for this type and size of engine. The AOAP Manual, Volume IV, actually specifies that the Cu concentrations can go up to 049 ppm before this wear is considered to be abnormal.

This considered opinion is based on the fact that there are several similar Cummins engines which are shown in the AOAP Manual, Vol. IV, which have a higher range of Copper (Cu) concentrations. For example, there is a similar Cummins engine Model NTC-290 (used in VEH F5070, a 20-ton dump), with Cu normal limits of 0 to 092 (ppm), and with a high limit for Cu shown at 115 to 142 (ppm). Another example evident is the Cummins Model NTC-400 engine (used in VEH M915A1, 10-ton tractor), with assigned Cu normal limits of 0-062 (ppm), and a high range of 077 to 095 (ppm). It should be mentioned that these specified Cu limits are based primarily on empirical data obtained from a history of military vehicles, as recorded in the AOAP files and records. Also, to reiterate, it is possible for the normal AOAP sampling data to incorporate (and exhibit) some Cu concentrations which were due in part to Cu additives which are used by some oil formulator companies as an anti oxidant agent. These Cu additives can sometimes result in AOAP lab results of Cu concentrations in the range of 040 to 100 ppm, but are normally in concentrations of less than 005 ppm. (See Table 5-4).

In general, the Fe concentrations in ppm were not found to be excessive for any of the subject (50) trucks. However, several of the trucks in the program did exhibit Fe concentrations in the range of 150 to 200 ppm, which were approaching the AOAP specified limit of 233 ppm.

5.1.4. CHANGES AND DEPLETIONS IN OIL ADDITIVES

During the initial 12 months of the oil filter program, the Zinc (Zn) ppm concentrations showed definite increases for all 50 of the M939A1 vehicles (i.e., "A", "B" and "C" battery trucks). The Zn additive is used as an anti oxidant and also as an anti wear agent. The Zn concentrations exhibited increases from initial values of 700 to 750 ppm, up to numbers which exceeded 1100 ppm. These increases were considered to be due, in part, to the fact that as new oil was added to the truck engines, the new oil had Zn additives in it, and these Zn additives become accumulative.

The Silicon (Si) additives remained fairly constant in the engine oils as the testing progressed. The Si additive is present as an anti foaming agent in the oil. Silicon can also accumulate in the engine oil due to dirt ingestion. During the subject testing, none of the test vehicles exhibited any Si readings which were in close proximity to the "AOAP" limits of 060 ppm.

The magnesium (Mg) concentrations, which were obtained during the two-year test program, exhibited some noticeable variations, or fluctuations up and down, throughout the test. These fluctuations were probably due to the varying composition of added make-up oil. However, when the test Mg numbers were viewed on an average basis, the Mg numbers remained relatively steady during the test. The additive component which contains Mg is used in engine oils as an acid neutralizer and a detergent for sludge and varnish. It also acts as a dispersant to hold the sludge and varnish in suspension in the engine oil. In addition to the Mg additive, calcium (Ca) is present in another additive which has the same function as the Mg additive. Overall, the calcium concentrations were observed to be in the range of approx. 1000 to 1200 ppm. In summary, the "by-pass" oil filters did not appear to have any effects on the zinc, magnesium, or calcium additives in the engine oil.

5.1.5. TAN AND TBN TEST RESULTS

When an engine oil is subjected to high temperatures, as well as normal operating conditions, for extended periods of time, several undesirable reactions occur in the oil. The anti-oxidant additives, which are there to protect the bulk of the oil, are gradually depleted. This occurs due to acids which are formed from incomplete combustion products and from some partial oxidation of the oil. These acids then react with the alkaline additives in the oil, which in turn reduces the basicity (TBN) of the oil. If not neutralized, these acids can attack the metal surfaces inside the engine (bearings, cams, etc.).

The test method employed to monitor the used oil for its total base number (TBN) is the ASTM D664 method (see Appendix B). The TBN is defined as "the quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, that is required to neutralize all basic constituents present in 1 gram of sample" (expressed in units of mg KOH/gram oil).

The normal TBN values in new MIL-L-2104 engine oil range from 6 to 12, and many used oil analysts recommend that the TBN number should not decrease below a value of 2.0 mg. KOH/gram oil. Also, the ASTM D664 method is used to monitor the relative rates of acid build-up in the used oils, which is recorded as an increase in the total acid number (TAN).

The normal TAN numbers in new MIL-L-2104 engine oil range from less than 1.0 to 3.0 mg KOH/gram oil, and many used-oil analysts recommend that the TAN should not exceed 5.0 mg KOH/gram oil.

NOTE: Monitoring of TAN and TBN values in used oil is considered to be an essential part of proper used oil analysis. The accurate monitoring of these values enables the evaluating technician to determine when the acids in used oil reach unacceptable levels. As is well known in engine-wear analysis, the presence of high acidity in engine oil is conducive to corrosion and wear on the metallic components in the engine.

The current standards for U.S. Army "AOAP" laboratory analysis do not include the ability to measure or record TAN and TBN values. It is the considered opinion of many oil analysis experts that the monitoring of these TAN & TBN values should be incorporated into the U.S. Army "AOAP" standards.

As can be seen in the data shown in Tables 5-1 through 5-3 for TAN and TBN values, there were no instances where the TAN values were greater than the guideline upper limit of 5.0 mg KØH/gm oil. However, there was one instance (see Table 5-2 - VEH. B-11), where the final reading (May 89) exhibited a TAN value of 4.7. But in this instance, the corresponding TBN value was 4.2 mg KØH/g, which was at an acceptable level and was (theoretically) able to counteract this relatively high TAN number.

A further review of the data shown in the three charts for TAN and TBN values, shows that there were no TBN numbers less than the lower guideline limit of 2.0 mg KØH/gm oil, except for the VEH A-21 (at Apr 89 sampling). In this instance the TBN value was at 1.5 mg KØH/gm. However, the corresponding TAN number was at 2.5, which was within the acceptable limits. And, as can be seen in the same chart, the subsequent readings for TAN & TBN for VEH A-21 were both within the guideline limits. Therefore, this instance of a low TBN value for this one vehicle was not considered to be of any serious consequence.

Overall, the by-pass oil filters did not appear to have any effect on TBN decrease or TAN increase during the test.

5.1.6. DR FERROGRAPH TEST RESULTS

As a prelude to this discussion, it is necessary to present here a definition of the terms which are used in the DR-Ferrograph test method. The Wear Particle Concentration (WPC) is the sum of the D_L and D_S particle counts. These terms are defined as follows: (Also see enclosure).

TABLE 5-1.

**"A" BATTERY VEHICLES
(TAN & TBN VALUES)**

END TEST

OIL SAMPLING DATES

VEH NO.	JULY 1988		SEPT 1988		NOV 1988		JAN 1989		APR 1989		MAY 1989	
	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN
A-01	3.3	3.4	DATA NOT AVAIL. " " " " " " " " " " " "		2.6	3.9	2.5	3.9	2.9	3.7	3.0	3.6
A-04	3.0	3.7			2.3	3.3	2.4	3.5	2.8	3.2	3.1	2.7
A-09	2.9	4.4			2.3	3.6	2.4	4.7	2.7	3.5	2.7	6.2
A-11	3.3	3.4			2.6	3.2	2.5	3.0	3.1	2.8	2.8	3.4
A-12 *							1.8	5.2	2.1	5.3	3.0	2.9
A-13	3.6	3.1			3.1	4.2	3.4	6.5	3.1	4.1	2.5	5.8
A-14	3.0	3.9			2.6	3.0	2.5	3.9	2.8	2.2	2.6	3.7
A-15	3.1	3.3			2.8	3.4	2.6	4.2	2.4	3.5	2.5	4.5
A-16	3.7	3.4			2.9	4.1	3.1	4.3	2.9	3.8	3.9	3.0
A-20	3.0	3.7			2.4	4.1	2.4	3.1	2.6	4.1	2.9	5.5
A-21	2.9	4.8			2.5	3.7	2.6	4.3	2.5	1.5**	3.3	3.3
A-22	2.9	4.7			2.3	3.6	2.6	4.8	2.4	3.3	3.0	3.5
A-23	2.9	5.3			2.4	3.7	2.6	5.8	2.3	3.5	2.6	3.2
SVC-20	3.0	3.7			2.6	3.8	3.0	5.6	2.6	4.3	2.5	3.9
SVC-21	3.1	2.8			2.6	3.2	2.7	3.6	3.2	3.3	3.1	3.5

** - Low Value

NOTE: These values are expressed in terms of mg KOH/gram oil

* Engine was replaced in Veh A-12 on 17 Aug 88

TABLE 5-2.

**"B" BATTERY VEHICLES
(TAN & TBN VALUES)**

OIL SAMPLING DATES

END TEST

VEH NO.	JULY 1988		SEPT 1988		NOV 1988		JAN 1989		APR 1989		MAY 1989	
	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN
B-01	3.3	5.3	DATA NOT AVAIL. " " " " " "		2.7	3.8	3.0	3.8	2.6	4.4	3.1	5.4
B-04	3.3	4.0			2.5	3.5	3.0	7.0	2.5	3.7	3.1	4.3
B-09	3.2	4.2			2.5	4.2	2.7	4.7	2.7	5.4	3.2	4.7
B-11	3.9	4.6			3.2	3.5	3.4	4.1	3.1	4.5	4.7 **	4.2**
B-12 *	2.8	7.6			2.2	6.1	2.7	5.7	3.4	6.9	3.0	6.7
B-13	3.7	3.9			2.9	3.3	3.0	5.2	2.9	4.9	3.8	5.4
B-14	3.5	4.1			2.5	3.5	2.9	3.7	3.0	4.3	4.5	3.4
B-15	3.4	4.6			2.7	3.8	3.1	6.1	2.9	5.5	3.5	4.4
B-16	3.4	4.1			2.7	3.1	2.7	5.5	2.7	4.8		
B-20	3.4	4.9	"		2.9	4.1	2.9	5.6	3.1	5.7	3.3	5.5
B-21	3.0	3.2	"		2.7	3.1	2.7	4.6	2.9	4.4	3.3	3.4
B-22	3.0	4.7	"		2.4	3.3	2.5	4.6	2.9	4.1	3.7	4.6
B-23	3.1	3.7	"		2.4	3.7	2.5	3.7	2.5	3.2	3.1	4.8
SVC-23			"		3.0	4.3	3.1	5.3	2.0	4.8	3.6	4.9
SVC-34	3.1	4.0	"		2.6	3.5	2.8	4.2	3.9	3.9	3.0	3.6

** - High Value

NOTE: These values are expressed in terms of mg KOH/gram oil

* Engine was replaced in Veh B-12 on 1 May 1988

TABLE 5-3.

"C" BATTERY VEHICLES
(TAN & TBN VALUES)

OIL SAMPLING DATES

END TEST

VEH NO.	JULY 1988		SEPT 1988		NOV 1988		JAN 1989		APR 1989		MAY 1989	
	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN	TAN	TBN
C-01	2.8	4.1	2.8	3.8	2.5	3.0	2.8	3.8	2.8	4.8	3.6	3.1
C-04	3.3	4.7	3.0	4.1	2.6	4.1	2.9	5.2	2.9	4.3	3.6	4.2
C-09	2.9	4.6	3.0	4.4	2.5	2.9	2.6	3.7	2.6	3.1	2.7	4.0
C-11	3.8	3.8	3.4	2.8	2.8	3.4	3.0	4.9	2.8	4.1	3.3	3.7
C-12	4.6	4.3	4.0	3.1	3.2	3.8	3.2	3.1	3.4	3.7	3.4	4.0
C-13	4.2	2.8	3.1	3.8	3.2	3.2	3.3	3.7	3.4	4.0	3.2	6.4
C-14	3.7	3.4	3.5	3.4	2.8	3.9	3.1	3.4	3.1	3.1	3.0	3.0
C-15	3.3	4.1	3.1	3.7	2.6	3.4	2.9	4.1	3.4	4.0	3.0	3.4
C-16	3.5	3.2	2.6	3.6	2.7	3.1	2.9	4.3	3.2	2.9	2.3	3.1
C-20	2.6	4.8	2.3	3.6	2.4	3.5	2.6	3.3	2.9	2.1	2.6	3.1
C-21	2.9	3.6	2.8	3.8	2.6	3.2	2.9	3.9	2.5	4.0	2.3	4.3
C-22	3.0	4.7	2.5	3.8	2.6	3.4	2.7	4.7	3.8	2.7	2.5	5.1
C-23	3.1	2.9	2.9	3.1	2.3	3.8	2.9	3.2	3.8	2.6	1.1*	3.3
SVC-24	3.3	3.8	2.8	2.8	2.6	3.6	3.1	3.7	2.2	5.7	2.5	4.4
SVC-25	3.7	4.0			3.2	4.1	3.0	6.0	2.4	6.1	2.6	6.0
SVC-26	3.4	4.4			2.6	3.8	3.2	4.9	2.3	3.9	2.3	4.0
SVC-27	2.8	3.9			2.3	4.1	2.7	5.1	2.3	3.1	2.8	3.9
SVC-28	3.1	3.9			2.4	3.2	2.8	4.5	2.3	4.4	2.2	3.7
SVC-29	2.9	4.1	2.6	3.2	2.3	4.6	2.6	4.4	2.9	4.4	2.8	4.9
SVC-30	2.9	6.2	2.3	6.4	2.4	5.5	2.8	5.0	2.5	6.5	2.6	6.3

NOTE: These values are expressed in terms of mg KOH/gram oil

*New Oil

TABLE 5-4. FT. LEWIS, WA LUBE OIL SAMPLES (NEW)

TABLE 5-4			
Ft. Lewis, WA Lube Oil Samples (New)			
AL Code	16207	16208	16209
Date Sampled	5-14-87	5-14-87	5-14-87
QPL No.	MC-2735	MC-3030	MC-2542
SAE Viscosity Grade	15-W40	15W-40	15W-40
17478			
3-15-88			
MC-2549			
15W-40			
Properties			
KVIS 40C	13.83	14.85	13.60
KVIS 100C	114.40	117.15	113.17
VI	120	130	118
Sulfated Ash %W	0.90	0.89	0.90
14.42			
109.99			
134			
ND			
Elements			
Sulfur %W	0.42	0.66	0.44
P	0.10	0.11	0.10
Ca	0.12	0.11	0.11
Ba	NIL	NIL	NIL
Zn	0.12	0.12	0.12
Na	NIL	NIL	NIL
N	0.04	0.03	0.04
Mg	0.05	0.04	0.05
B	NIL	NIL	NIL
Cu	NIL	NIL	NIL
0.55			
0.11			
NIL			
NIL			
0.11			
ND			
0.06			
0.12			
158			
3			
TAN	2.5	2.6	2.5
TBN D664	4.9	4.1	4.7
3.6			
8.3			

ND = Not Determined

NOTE: Two 15W40 oils (source samples) were received from Ft. Lewis in May, 1989. They were analyzed for copper content with the following results:

Sample No.	MC-	CU ppm
AL-18738	2694	10
AL-18739	Unknown	54

D_L = The collection (count) of ferromagnetic particles which are of 5 μm size and larger which are deposited within a few millimeters after entering the magnetic field of monitoring device, and this collection is called the "entrance deposit".

D_S = The next collection of ferromagnetic particles, which are smaller than 5 μm in size, which are deposited at a point about five millimeters (.195 in) downstream from the "entrance deposit," this collection is called the S deposit.

As can be seen in the results of the DR - Ferrograph tests (Table 3-1), the summation of the D_L and D_S values, or the WPC value, always exhibited a larger value for the D_L figure, than for the D_S value. And, since the D_L value represents ferromagnetic particles which are larger than 5 μm in size, these particles would indicate a potential wear-damage factor for engine components. This is because it is generally considered by analysts for engine wear that metal particulates of less than 5 μm size, do not present a serious threat to engine wear. However, it should be mentioned that, quantitatively speaking, the actual effects on wear of these 5 μm and smaller particles are not precisely known at this time. Also, it should be mentioned that these military M939A1 vehicle engines were not given any tear-down inspections after the completion of the two-year program. It was not feasible to conduct any tear-down of these engines, since these military trucks were being utilized for continuing Fleet Operations by the 9th I.D. at Fort Lewis, Wash.

In Table 3-1, the WPC values shown for the "A" and "B" Battery vehicles, were lower in all cases than the WPC values shown for the "C" Battery vehicles. Also, it can be seen that these WPC values for the "A" & "B" Battery trucks were less than the "C" Battery trucks, even when the 14.8 percent oil dilution factor is taken into account. For example, the WPC values shown in Table 3-1 for the sampling #7 (at 12-month period), show a WPC of 25.3 for "A" Battery, and 22.9 for the "B" Battery, as compared to a WPC of 40.4 for the "C" Battery trucks. It can be easily seen that a 14.8% increase in either the "A" or "B" Battery values (approx. 3.5 increase), would not alter this relationship appreciably. The only exception to this relationship was found in the final sampling (No. 13), where the WPC value for the "A" Battery of 41.5 when increased by 14.8 percent, would be slightly above the "C" Battery WPC value of 46.9. (See Table 3-1)

5.2 LAB TEST RESULTS PER SAE HS-J806b SPECIFICATION

5.2.1 DISCUSSION

The two types of "by-pass" oil filters, Filter "A" and Filter "B", were tested at the Southwest Research Institute laboratories per the requirements of SAE HS-J806b, and the Chapters 1, 2, 3, 4 and 7.

A comparison of the laboratory test results for the two subject filters is presented as follows:

CHAPTER 1 - Resistance to Flow

As was noted in the laboratory report, the Filter "B" exhibited a lower resistance to flow (psi) than did the Filter "A" for all flow rates below 1.00 GPM.

CHAPTER 2 - Filter Capacity and Contaminant Removal Characteristics

The Filter "B" showed a higher capacity in grams (14.72 gms) than did the Filter "A" (11.43 gms), at a point where 50% of the original flow through filters was attained, and the Filter "B" showed greater life in hours.

CHAPTER 3 - Single Pass Particle Retention Capacity

As was shown in the laboratory test results, the Filter "A" exhibited a single pass efficiency of 99.1 percent, as compared to the Filter "B" efficiency of 79.1 percent. However, it should be mentioned that these results could be somewhat misleading, since this particular test was designed primarily for full-flow type oil filters, which have much higher flow rates than do the bypass oil filters.

CHAPTER 4 - Media Migration Test

The test results showed relatively low rates of media migration for both subject filters, with the Filter "B" exhibiting a media migration rate approximately only 1/3 of that for the Filter "A".

CHAPTER 7 - Ability to Meet Environmental Conditions

For the effect of water in the oil on the filter contaminant capacity, the capacity in grams of AC dust was nearly

identical for both subject filters, and the Filter "B" exhibited a somewhat longer life in hours. However, this could be expected since the interior volume of the Filter "B" housing (approx 3.25 qts) is slightly larger than the Filter "A" housing (approx. 2.75 qts). Also, it was noted that the test results showed that neither of the subject filters affected the additive contents of the oils used for the tests.

CONCLUSIONS

The overall comparison of these test results would indicate that the oil Filter "B" exhibited slightly better filtration capabilities (in some tests) than the Filter "A". One difference that was shown was in the resistance to flow (chap. 1), where the Filter "B" displayed slightly less pressure resistance than did the Filter "A". However, it should be mentioned here that previous lab testing has shown that, for any given filter type, the filters internal resistance to flow may vary by as much as ± 1 psi, for a given flow rate.

In the single pass particle retention capacity test (chap. 3), it can be seen that the Filter "A" exhibited a better single-pass efficiency. However, this particular test is not considered to be very meaningful for by-pass type filters, due to their very low flow rates, as compared to the standard full-flow filters. This comparison between the two subject by-pass oil filters is better shown in the Chap. 2 test, which considers the overall filter capacity and contaminant removal characteristics. In this test, the Filter "B" exhibited a higher capacity in grams (14.72 gms) than did the Filter "A" - (11.43 gms).

5.3 COST EFFECTIVENESS COMPARISON ANALYSIS

5.3.1 In comparison of the various cost factors associated with the installation and usage of by-pass oil filters on military vehicle engines, there are the following factors to be considered:

The unit costs involved with each type of by-pass oil filter (as tested at Fort Lewis, Wash.) and shown in the Table 5.5., reveal that the basic unit costs are variable, and relatively high. As can be seen in the chart, the Filter "A" + kit costs \$293.00, as compared to a total cost of \$155.00 for the Filter "B" Assembly. It should be mentioned that these unit costs were supplied by the respective oil-filter assembly manufacturers, and these unit prices can change at any time, like any other NDI commercial item.

Another factor to be considered is the labor cost for installation of these by-pass oil filters on the military vehicle engines. The labor cost, as determined by the man-hours used to install these oil filters on M939A1 trucks at Fort Lewis, WA, was approximately \$87.00 per unit. This is based on the average time required to install these by-pass filters, based on U. S. Army Maintenance man-hours (3 hours at \$29.00 hr). Of course, this labor cost factor would be changed appreciable (presumable lower) in the event of a factory-installed oil filter. There are no cost factors available for a factory installation at this time.

An evaluation of the two-year oil-filter program at Fort Lewis, WA, indicated that the best possible projected cost savings which could occur due to usage of the by-pass oil filters would be (in some cases, not for all vehicles); the elimination of less than one-half (1/2) of one (1) engine oil change, in a two-year period. This would be an actual cost savings of less than \$48.00 per vehicle, based on Army Maintenance costs per oil change as follows: 27 qts of MIL-L-2104 oil at \$1.25/qt (\$33.75), and (1) filter element kit (\$33.00), plus one hour of labor at \$29.00. (Total cost of \$95.75).

5.4. COST ANALYSIS RESULTS.

Based on the cost figures shown for a two-year period, that is, the total cost of the installation of by-pass oil filter, and the savings of less than 1/2 of oil change, this is not a cost-effective program. However, since the two-year test program at Fort Lewis, WA, did not allow for any evaluation of long-term engine-wear characteristics on these truck engines, i.e., for operation with and without by-pass oil filters, this question, or test parameter, cannot be answered. But, it can be stated here that since the subject military trucks (M939A1's) accumulate much less mileage than similar commercial trucks (5-ton), say, in a 10 year period, the effects of oil lubrication on engine wear may not be a serious consideration.

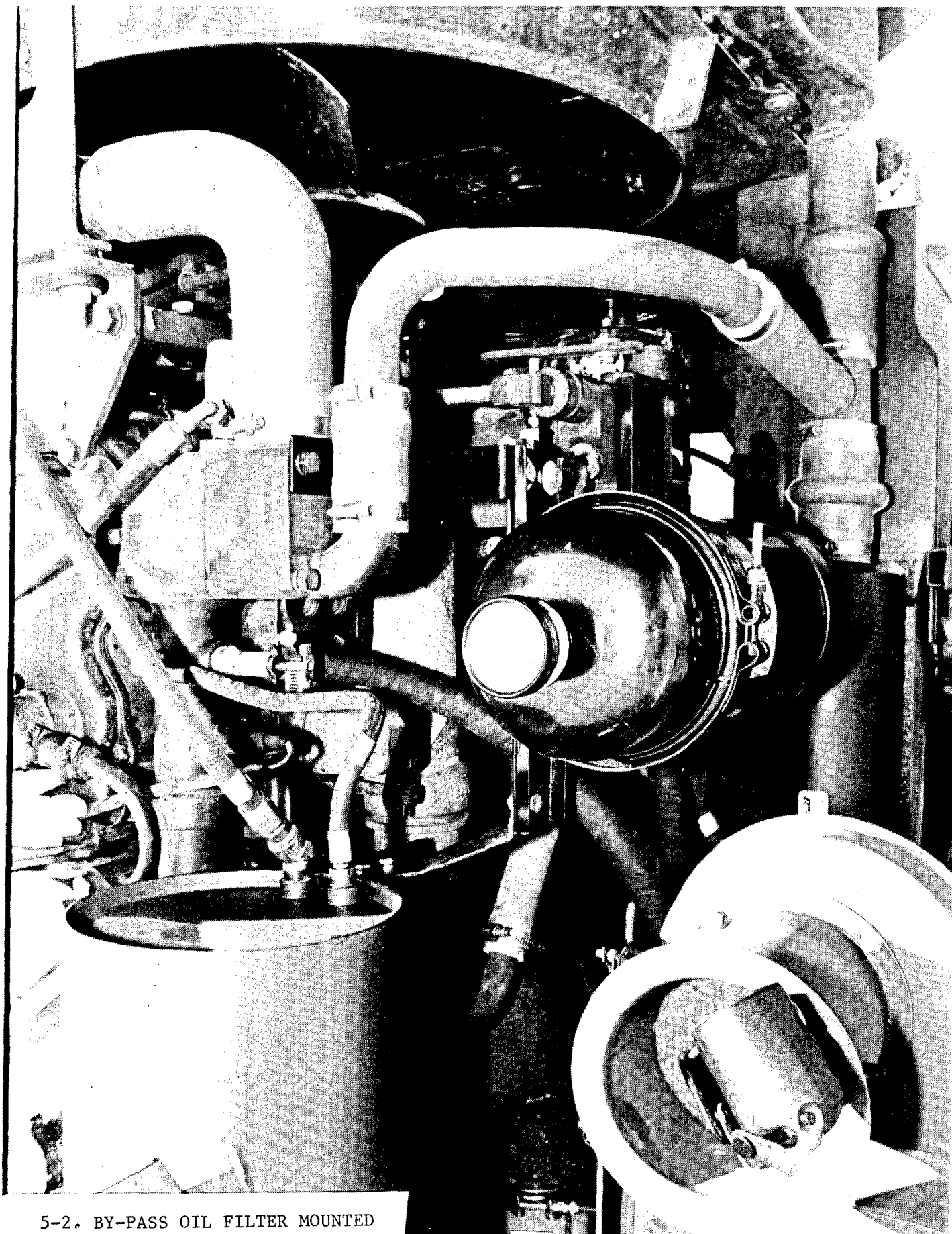
5.5 TEST PROGRAM PROCEDURES

5.5.1 INSTALLATION OF "BY-PASS" OIL FILTERS

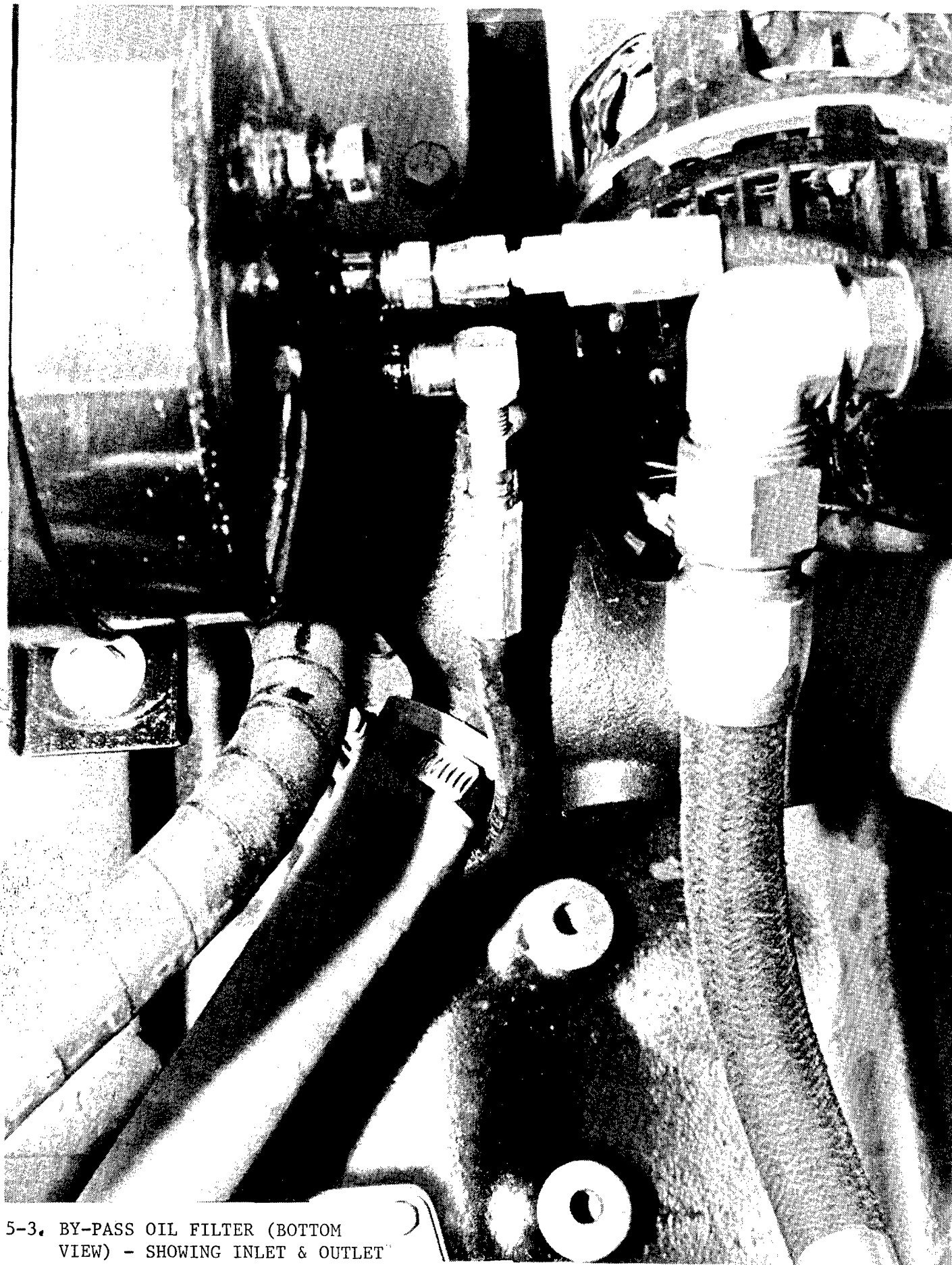
There was a total of 30 by-pass oil filters installed (15 ea. from two suppliers). (Filter "A" and Filter "B"). (See Figs. 5-2. thru 5-9.).

(1) These filters were installed by two teams of technicians: two men from TACOM and two men from Fort Lewis (DS Level Maintenance men). Each oil-filter assembly will have a test serial number stenciled on HSG.

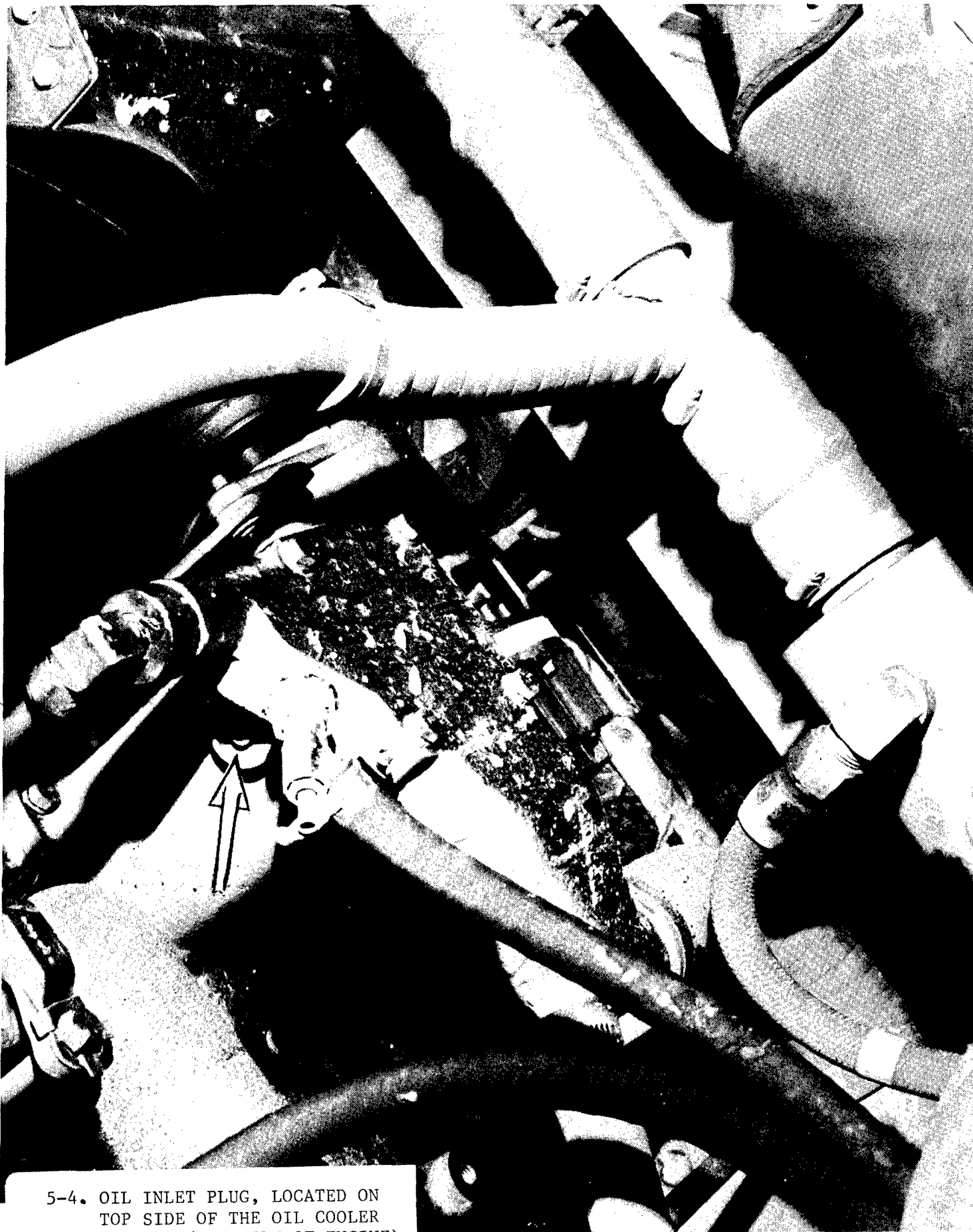
(2) Each fleet vehicle that was a part of test program, including 20 M939 trucks which were control vehicles, had their engine oil and oil filters changed at start of test.



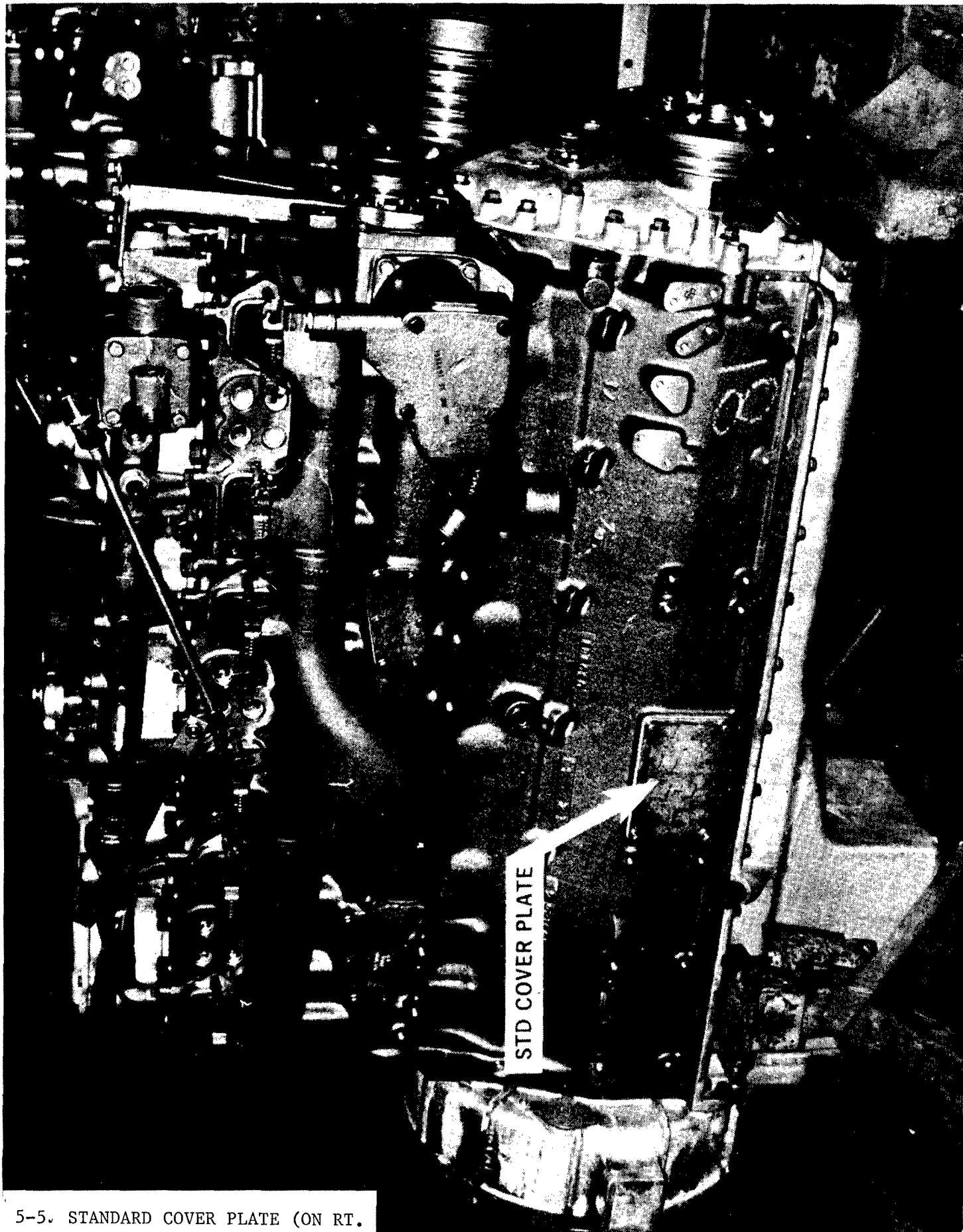
5-2. BY-PASS OIL FILTER MOUNTED
ON RT. SIDE OF ENGINE



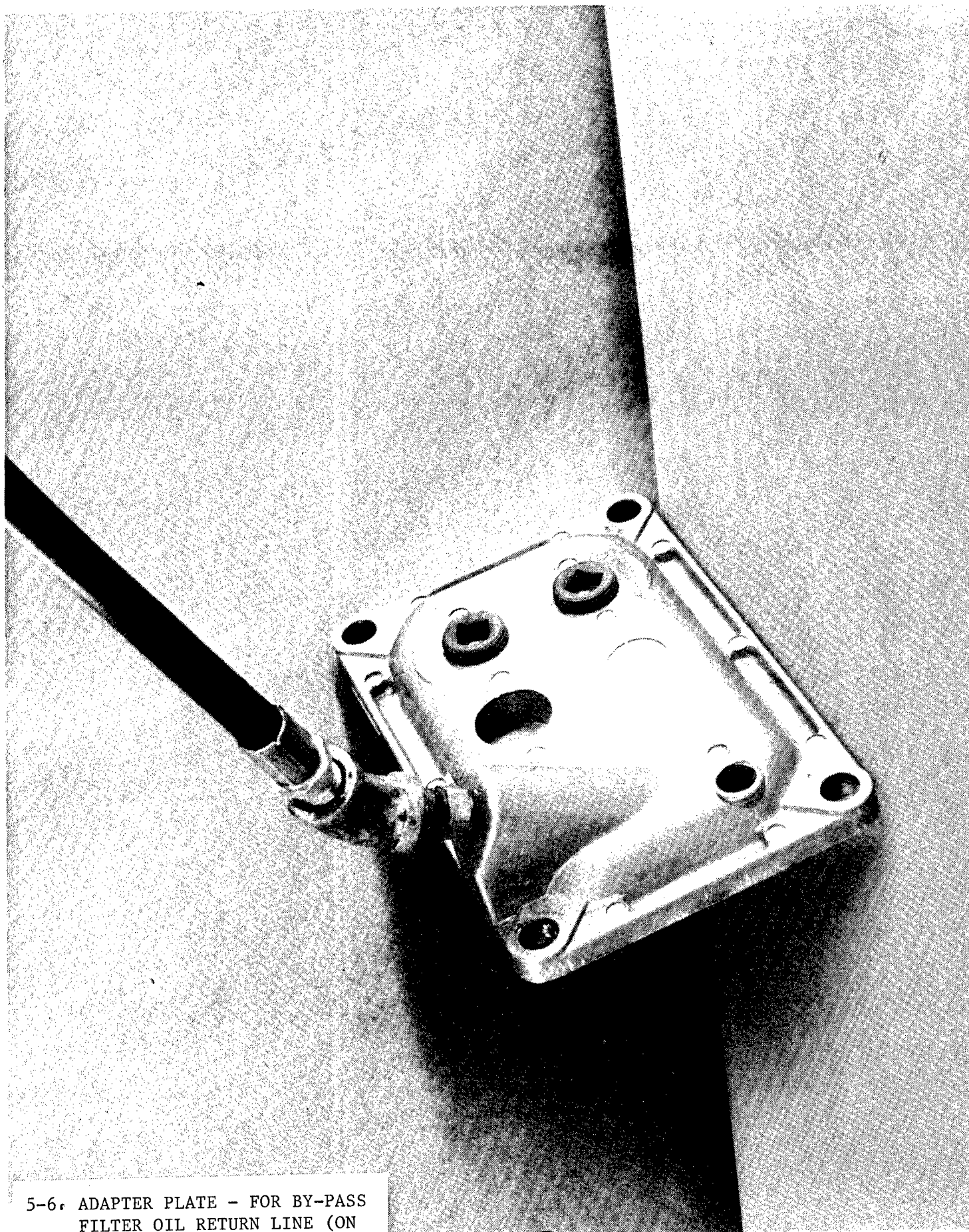
5-3. BY-PASS OIL FILTER (BOTTOM
VIEW) - SHOWING INLET & OUTLET
LINES



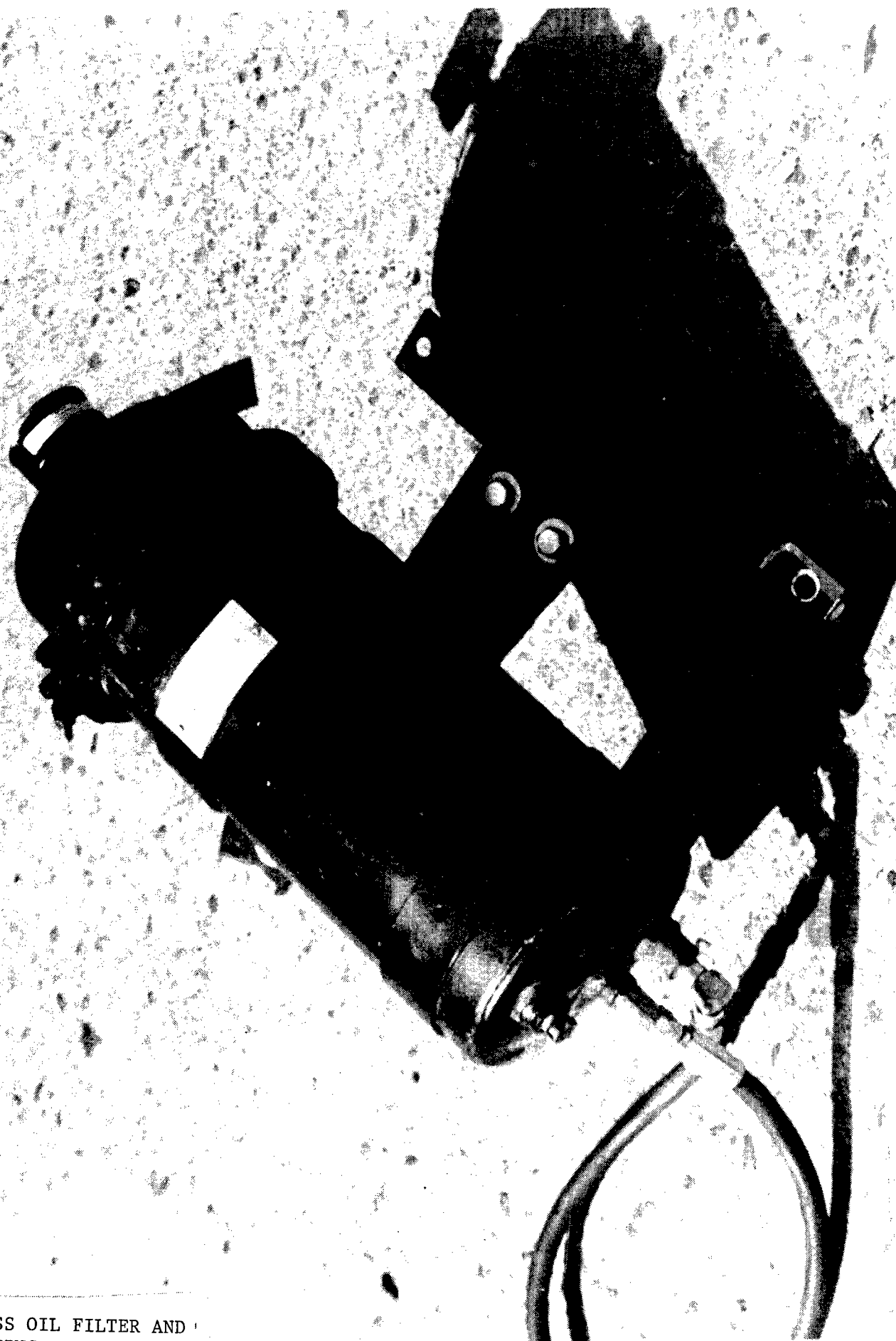
5-4. OIL INLET PLUG, LOCATED ON
TOP SIDE OF THE OIL COOLER
HOUSING (RT. SIDE OF ENGINE)



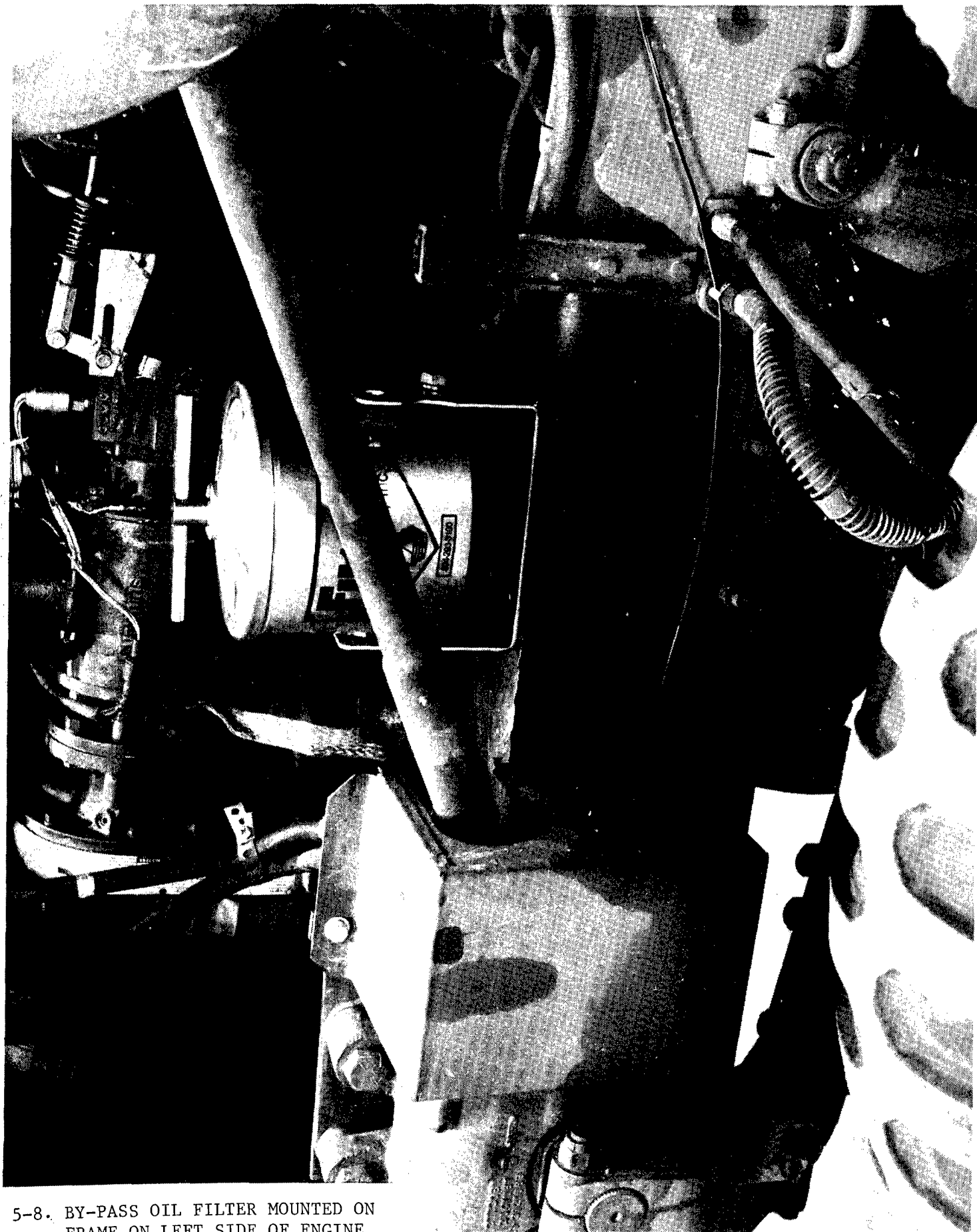
5-5. STANDARD COVER PLATE (ON RT.
SIDE OF BLOCK)



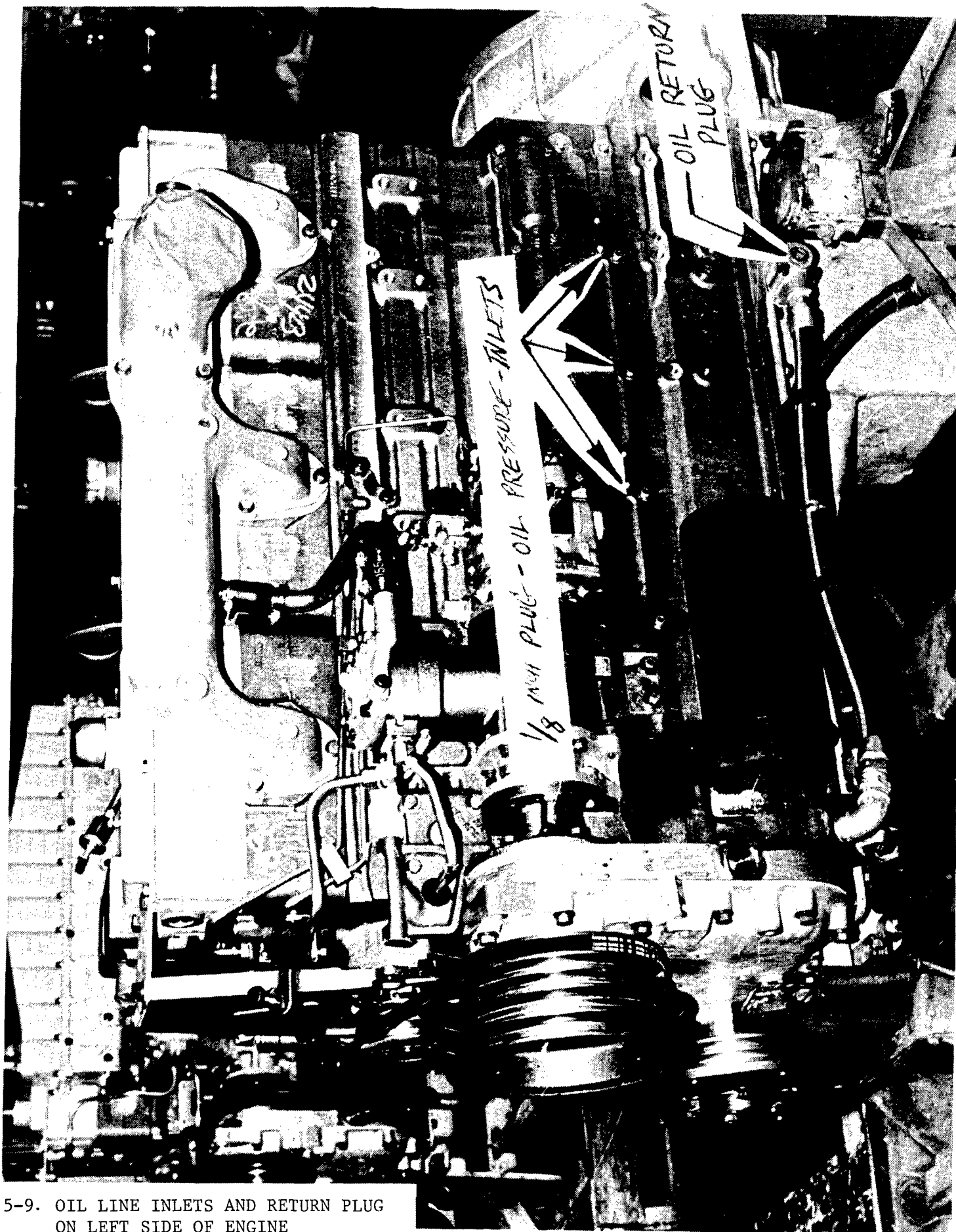
5-6. ADAPTER PLATE - FOR BY-PASS
FILTER OIL RETURN LINE (ON
RT. SIDE OF BLOCK)



5-7. BY-PASS OIL FILTER AND
ATTACHING BRACKETS



5-8. BY-PASS OIL FILTER MOUNTED ON
FRAME ON LEFT SIDE OF ENGINE



5-9. OIL LINE INLETS AND RETURN PLUG
ON LEFT SIDE OF ENGINE

(3) The oil used in these vehicles was SAE 15W40 (per MIL-L-2104) - Please record the supply oil drum qualification number.

(4) Each fleet vehicle in this test program had a specially marked Log Book (stored in the truck cab) - which incorporated log data entries as follows:

(a) Odometer reading & engine hours + date at start.

(b) Serial No. of the "by-pass" oil filter.

(c) Odometer reading & engine hrs + date - ea. time "AOAP" sample is taken.

(d) Odometer reading & engine hrs + date - ea. time oil is added.

(e) Odometer reading & engine hrs + date when oil is changed.

(f) Document any problems in vehicle engine operation (date, time odom/eng hrs).

(g) Document environmental conditions during vehicle operation (date, time, odom/eng hrs).

(h) Tagged selected oil filter elements when removed from test vehicles and held these filter elements for TACOM. (Identify by Tag No.).

5.5.2 "AOAP" SAMPLE COLLECTION DURING TEST PROGRAM

a. Frequency of two (2) oil samples on each vehicle

(1) Two oil "AOAP" samples taken each 60 days.

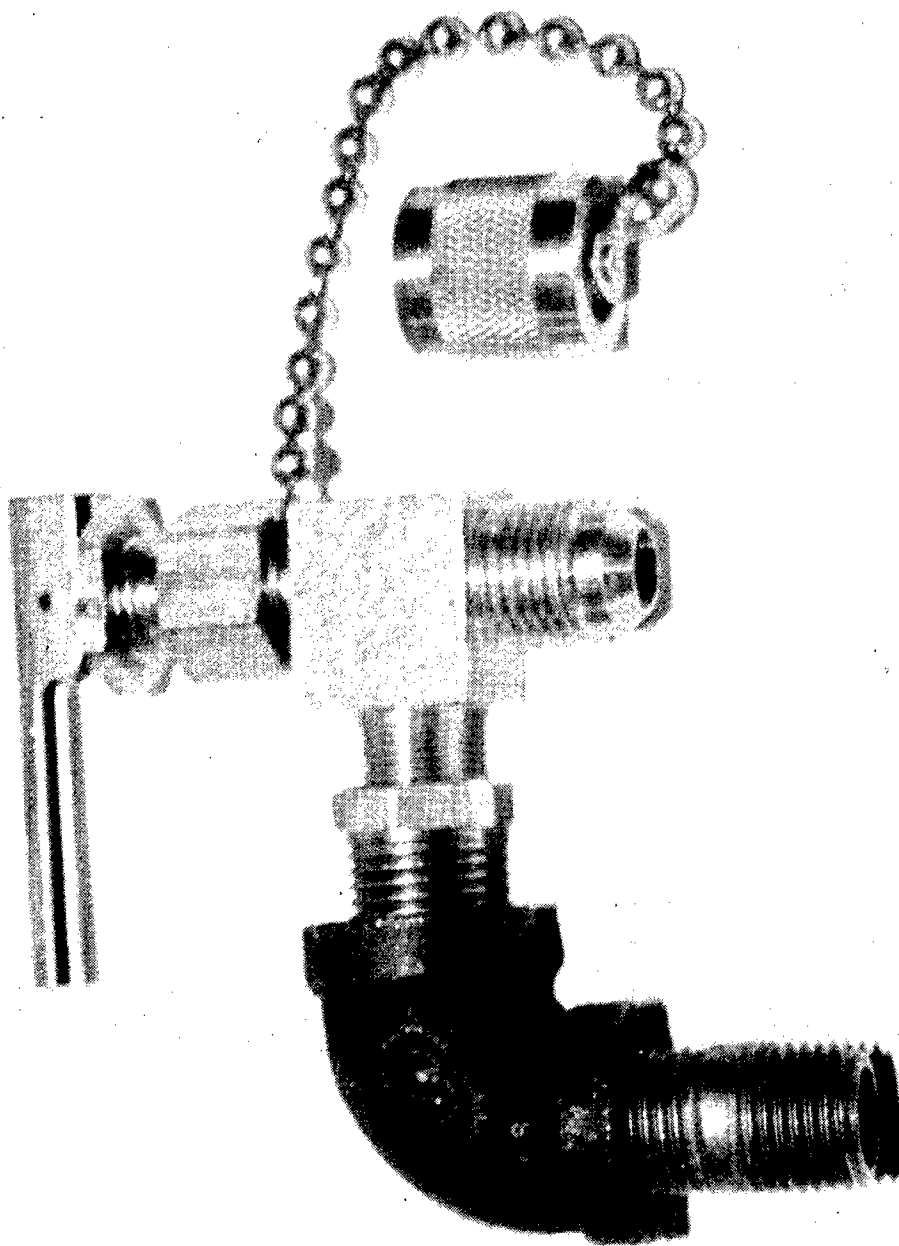
(2) Two oil samples taken simultaneously from each test vehicle. Oil samples were drawn out through a leak-proof valve (see Fig. 5-10).

b. Where "AOAP" oil samples were sent to:

(1) First oil sample sent to standard "AOAP" lab at Fort Lewis.

(2) Second oil sample was specially packaged and mailed to "BFLRF" lab at San Antonio, TX, 78284.

The AOAP sample bottles mailed to "BFLRF" were color coded yellow.



5-10. "FAIL-SAFE" VALVE
(NSN 2910-01-073-0080)

c. Whenever any amount of new oil was added to the engine on any test vehicle, the exact quantity of oil and date of oil addition was recorded in the vehicle log book on Form DD-2026.

d. There was not any changing of the test vehicle oil filter elements either full-flow or "by-pass" type filter elements, without the express approval of the Fort Lewis "Cobro" (Cook Brown Co.) support monitor. (See Figs. 5-11, 5-12, 5-13).

5.5.3 DATA COLLECTION & PROCESSING

a. The Fort Lewis "Cobro" support monitor for the oil filter test was responsible for monitoring (and collecting) the following data:

(1) He made certain that the vehicle log book driver entries were being made in satisfactory manner and regular basis on each test & control vehicle.

(2) He made certain that the two oil samples were taken on each vehicle at 60 day intervals.

(3) He recorded the oil drum qualification number from each new supply drum.

(4) He delivered the first AOAP sample to Fort Lewis lab, and second AOAP sample to Belvoir Fuels & Lubricants Research Facility, San Antonio, TX.

(5) Details for used oil analysis at BFLRF lab:

(a) Metallic concentrations (12 elements).

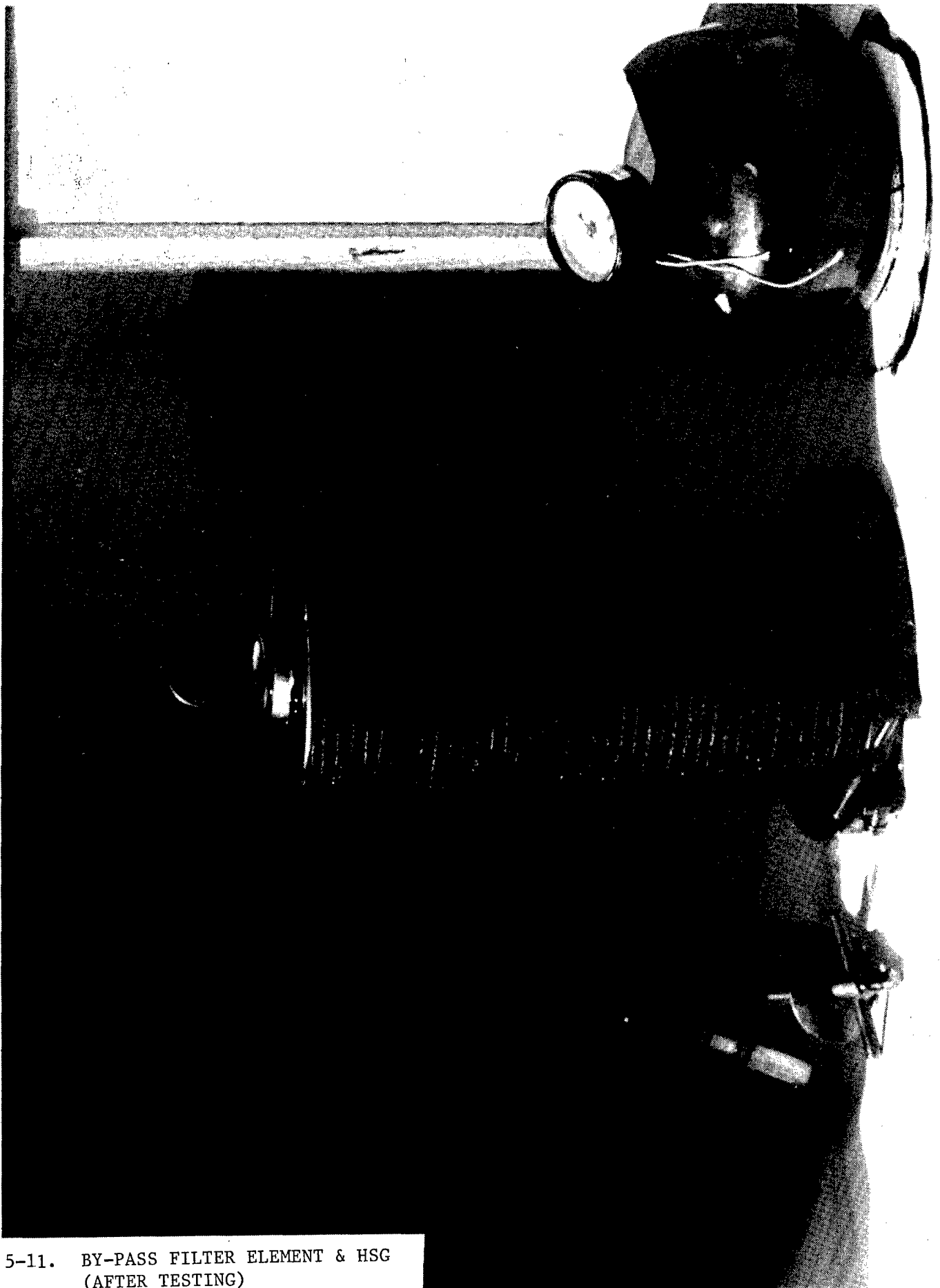
(b) Viscosity measurements.

(c) Record TAN & TBN values.

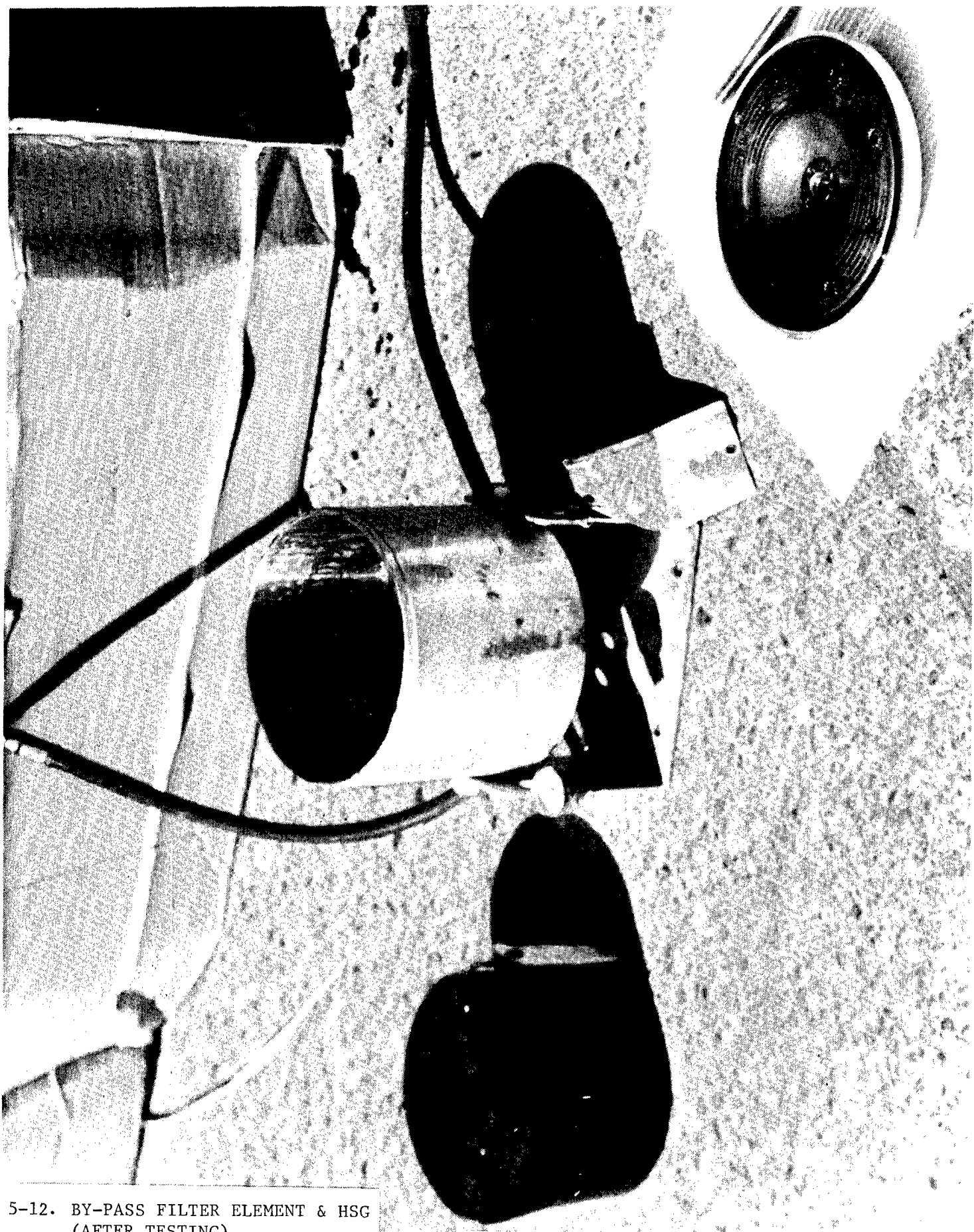
(d) Check for fuel dilution.

(e) Ferrographic tests.

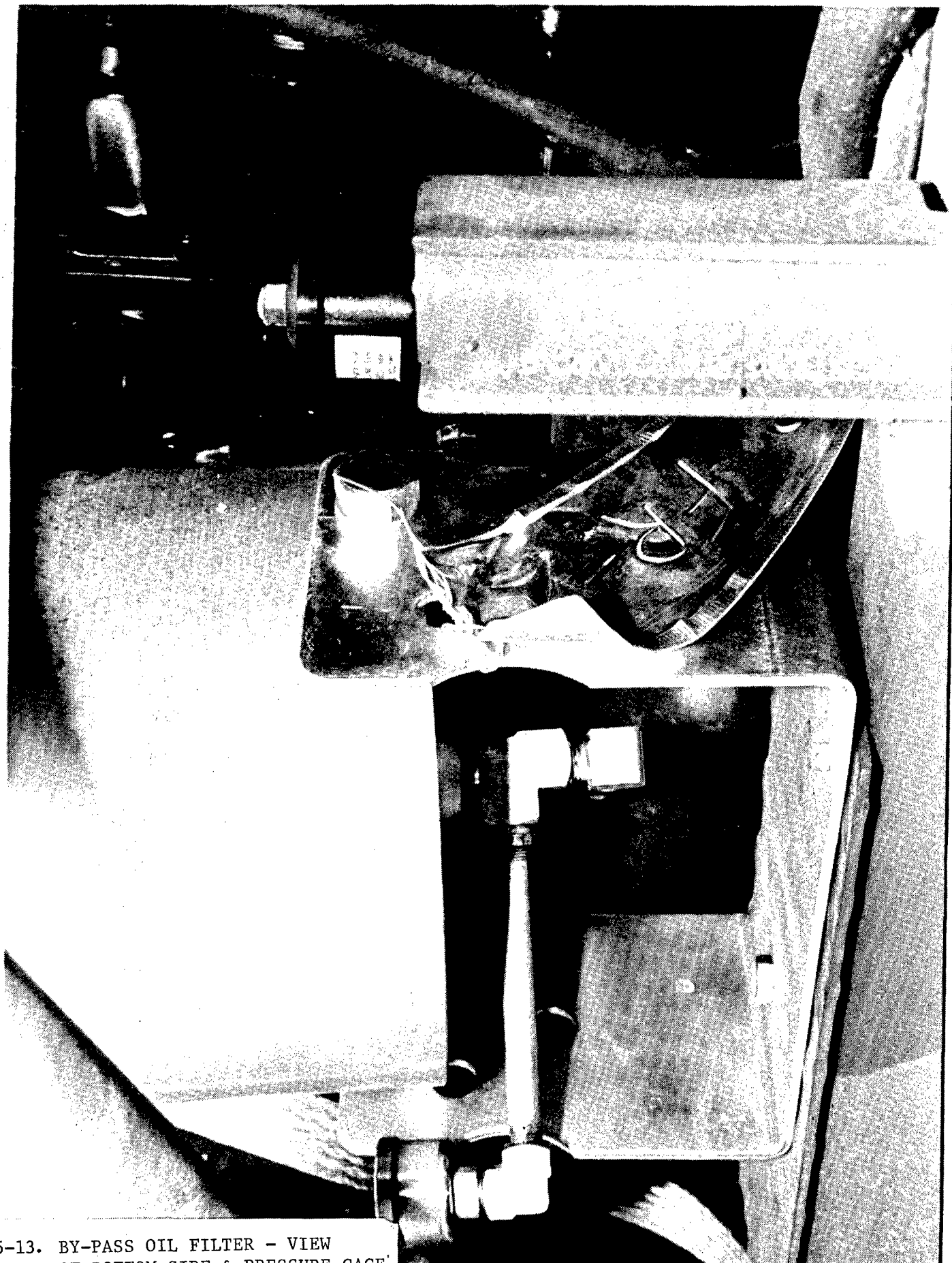
NOTE: After the completion of the two-year test program, approximately 40 gallons of used oil was collected from vehicles in each group ("A", "B" & "C" batteries), and this oil was shipped to BFLRF (at San Antonio, TX) for further laboratory tests. (See Fig. 5-14)



5-11. BY-PASS FILTER ELEMENT & HSG
(AFTER TESTING)



5-12. BY-PASS FILTER ELEMENT & HSG
(AFTER TESTING)



5-13. BY-PASS OIL FILTER - VIEW
OF BOTTOM SIDE & PRESSURE GAGE



5-14. DRUMS OF USED OIL (AFTER COMPLETION OF TESTING TO BE SHIPPED TO BFLRF LAB, SAN ANTONIO, TX, FOR FURTHER TESTING)

5.6 SOURCE SAMPLES (NEW OIL AT FORT LEWIS, WA)

The engine oil used throughout the by-pass oil filter evaluation test was an uncontrolled variable. Supply oil in stock (per MIL-L-2104) at Ft. Lewis was used. Table 5-4 presents the inspection properties of four different source samples. Oils AL-16207, 16208 and 16209 were used when the by-pass filters were installed in May 1987. Sample AL-17478 was taken in March, 1988. Oils AL-16207, 16208 and 16209 were from different suppliers; however, they contained similar additive element contents. Oil AL-17478 had a different additive composition and did not contain calcium and also had a much higher magnesium content.

TABLE 5-5.

BASIC COST OF BY-PASS OIL FILTERS
COMPARISON FILTER "A" VS FILTER "B"

DESCRIPTION OF ITEM	UNIT COST OF ITEMS	
	FILTER "A" MODEL	FILTER "B" MODEL
C-100 By-Pass Filter Assy: 1 to 100 units	\$275.00 (ea)	
101 to 1000 units	233.75 (ea)	
Above 1000 units	220.00 (ea)	
Filter Element Replacement	18.00 (ea)	
Installation Kit:	55.00 (ea)	
(Hoses, fittings, adapters, & gauge)		
Labor (approx 3 man-hrs at \$29.00/hr)	87.00	
W/O Labor (lowest unit cost)		
GRAND TOTAL →	\$293.00	
BPT 5012 - Filter Assy 1 to 100		\$82.00
(Above 1000 units, a 2% discount)		
Filter Element - Replacement		15.00
Installation Kit:		
Hoses, fittings, adapters		58.00
Labor (approx. 3 man-hrs at \$29.00/hr)	87.00	
Lowest Unit Cost) GRAND TOTAL - W/O Labor →		\$155.00

TABLE 5-6. TACOM OIL FILTER PROGRAM MILEAGES

"A" FILTERS		TACOM OIL FILTER PROGRAM MILEAGES									
		VEH. NO.	COMPONENT NR	END ITEM SER#	ENTERED PROGRAM			EXITED PROGRAM			
					DATE	HOURS	MILEAGE	DATE	HOURS	MILEAGE	
	111330015	A-1	C523-09193	87135	54	975	89151	335	6296		
	111322621	A-4	C523-08935	87135	78	1437	89151	264	5242		
	111339342	A-20	C525-03010	87135	42	749	89151	284	4936		
	111344186	A-11	C525-02935	87135	62	1095	89151	390	6373		
	111362632	A-12 *	C525-03092	88255	202	3191	89151	242	3854		
	111338406	A-13	C525-03029	87135	84	1020	89151	370	6168		
	111337148	A-14	C525-03006	87135	50	776	89151	327	5892		
	111339329	A-15	C525-03105	87135	72	992	89151	352	4993		
	111333463	A-16	C525-02788	87135	60	988	89151	366	5920		
	111337933	A-9	C525-02778	87135	11	188	89151	221	3174		
	111331005	A-21	C523-09143	87135	36	461	89151	257	4758		
	111314837	A-22	C523-08823	87135	13	114	89151	311	3388		
	111322331	A-23	C525-08735	87135	14	139	89151	174	2678		
	111324506	SV-21	C523-08867	87135	64	1432	89151	434	9760		
	111339529	SV-20	C525-03084	87135	160	1425	89151	496	7080		
	111340268	A-12*	C525-03092	87135	69	1101	88254	202	3191		

* Engine was replaced in Veh A-12 on 17 Aug 88 Average Mileage = 4376.0 miles

Average Veh. Speed - 17.20 MPH

TABLE 5-7. TACOM OIL FILTER PROGRAM MILEAGES

"B" FILTERS		TACOM OIL FILTER PROGRAM MILEAGES							
		VEH. NO.	END ITEM SER#	ENTERED PROGRAM			EXITED PROGRAM		
COMPONENT NR	DATE			HOURS	MILEAGE	DATE	HOURS	MILEAGE	
11321356	B-1	C523-08659	87135	15	171	89151	156	2643	
11312949	B-4	C523-08866	87135	131	2658	89151	371	8071	
11344671	B-9	C525-03015	87135	73	927	89151	304	4573	
11340499	B-11	C525-03085	87135	83	1044	89151	415	6087	
11158628	B-12 *	C525-03039	88122	182	3259	89151	265	4396	
11331712	B-13	C525-02783	87135	73	1047	89151	393	6106	
11339339	B-14	C525-03051	87135	71	939	89151	421	4992	
11336186	B-15	C525-02803	87135	82	797	89151	454	4669	
11334745	B-16	C525-02796	87135	111	1100	89151	406	4428	
11319096	B-20	C523-08902	87135	23	417	89151	304	4279	
11311980	B-21	C523-08863	87135	81	1509	89151	394	8405	
11315847	B-22	C523-08839	87135	14	206	89151	273	4626	
11337404	B-23	C525-02792	87135	12	196	89151	290	6187	
11319091	SV-23	C523-08676	87135	94	1881	89151	439	8762	
11321035	SV-34	C523-08717	87135	90	1509	89151	433	9129	
11342084	B-12*	C525-03039	87135	89	1339	88122	182	3259	

* Engine was replaced in Veh B-12 on 1 May 88

Average Mileage = 4646.8

Average Veh. Speed = 16.7 MPH

TABLE 5-8. TACOM OIL FILTER PROGRAM MILEAGES

CONTROL VEHICLES		TACOM OIL FILTER PROGRAM MILEAGES					
COMPONENT NR	VEH. NO.	END ITEM SER#	ENTERED PROGRAM			EXITED PROGRAM	
			DATE	HOURS	MILEAGE	DATE	HOURS MILEAGE
11332216	C-1	C523-09232	87135	19	326	89151	294 5527
11326721	C-4	C523-09061	87135	107	1969	89151	383 7839
11344464	C-9	C525-02922	87135	50	822	89151	262 4469
11341736	C-11	C525-03055	87135	97	883	89151	385 5390
11341881	C-12	C525-03047	87135	69	955	89151	355 5862
11339968	C-13	C525-03106	87135	76	859	89151	384 5953
11338880	C-14	C525-03005	87135	80	939	89151	425 4795
11333939	C-23	C525-02797	87135	73	1216	89151	296 4527
11335956	C-16	C525-02779	87135	75	780	89151	360 5552
11322049	C-20	C523-08855	87135	70	1132	89151	286 5502
11314533	C-21	C523-08752	87135	82	903	89151	388 6384
11319327	C-22	C523-08704	87135	29	274	89151	324 6717
11341898	C-15	C525-03037	87135	5	14	89151	323 4674
11319785	SV-24	C523-08705	87135	96	1820	89151	403 7106
11318462	SV-25	C523-08667	87135	97	1781	89151	490 10662
11339344	SV-26	C525-03056	87135	47	896	89151	431 5652
11323610	SV-27	C523-08900	87135	39	671	89151	274 4101
11324788	SV-28	C523-08876	87135	25	270	89151	340 6754
11322633	SV-29	C523-08747	87135	47	752	89151	240 5427
11320727	SV-30	C523-08905	87135	39	668	89151	340 7437

Average Veh. Speed = 17.9 MPH

Average Mileage = 5121.4

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APPENDIX A

QUANTITATIVE FERROGRAPHY -
ITS MEANING AND APPLICATION

QUANTITATIVE FERROGRAPHY - ITS MEANING AND APPLICATION

The DR Ferrograph measures the concentration of wear particles in lubricating oil. This technical information note describes the quantitative techniques used, explains the significance of wear particles and wear particle equilibrium, and considers sampling strategy.

QUANTITATIVE MEASUREMENTS IN FERROGRAPHY

The DR Ferrograph separates out particles having positive magnetic susceptibility by means of a high gradient magnetic field. Magnetic separation is nearly 100% effective for ferromagnetic particles larger than 0.1 micrometers. The ferrographic separation technique causes all ferromagnetic debris larger than 5 micrometers to deposit a few millimeters after entering the magnetic field. Consequently, the "entrance deposit" contains all the larger particles and a representative portion of the smaller particles.

No ferromagnetic particles larger than a few micrometers penetrate further than a few millimeters downstream from the entrance deposit. Particle size becomes progressively smaller along the deposition path.

The DR Ferrograph senses particles at two locations. First, at the entrance deposit, usually referred to as "L" and second, about five millimeters downstream, usually referred to as "S". The DR Ferrograph senses the presence of particles by measuring the amount of light attenuated at the two deposition locations.

Wear Particle Concentration (WPC) is the sum of L + S, divided by sample size (volume). In most cases the sample volume is one ml, so WPC is simply L + S. "Percent Large Particles" (PLP) is calculated as follows:

$$\text{PERCENTAGE LARGE PARTICLES (PLP)} = 100(L - S)/(L + S)$$

When using the DR Ferrograph, the user may calculate WPC and PLP from L and S. These calculations are normally used for trending wear conditions and establishing quantitative baselines. See Examples A and B.

Example A:

Determine WPC and PLP when L readout is 12 and S is 6; sample volume 3 ml.

$$\text{WPC} = \frac{L + S}{\text{Sample Size}} = \frac{12 + 6}{3} = 6$$

$$\text{PLP} = \frac{L - S}{L + S} \times 100 = \frac{(12 - 6)}{(12 + 6)} \times 100 = 33\%$$

Example B:

One ml of sample is mixed with 4 ml of clean oil to produce a 5:1 dilution. One ml of this diluted sample is processed. Dilution is 5 to 1; therefore, sample volume is 0.2 ml.

$$L = 21$$

$$S = 12$$

$$\text{WPC} = \frac{L + S}{\text{Sample Size}} = \frac{21 + 12}{0.2} = \frac{33}{0.2} = 165$$

$$\text{PLP} = \frac{L - S}{L + S} \times 100 = \frac{(21 - 12)}{(21 + 12)} \times 100 = \frac{9}{33} \times 100$$

$$\text{PLP} = 27\%$$

Other combinations of L and S have been tried by ferrograph users for trending data, but WPC and PLP are recommended because they relate to concentration and size distribution clearly and directly.

See Appendix I, Establishing Quantitative Baselines with the DR Ferrograph.

SIGNIFICANCE OF WEAR PARTICLES

Concentration and size distribution of wear particles reveal considerable information about the condition of lubricated wearing surfaces within a machine. Figure 1 illustrates how particle concentration and size increase as the wear process progresses from a normal operating condition, to incipient failure and, finally, to catastrophic failure. It should be understood that each failure mode (cutting wear, fatigue, etc.) exhibits a different failure profile and Figure 1 is merely a typical example of a wear condition.

APPENDIX B

STANDARD TEST METHOD FOR ACID NUMBER OF PETROLEUM
PRODUCTS BY POTENTIOMETRIC TITRATION



Designation: D 664 - 89

APPENDIX B

An American National Standard
British Standard 4457



Designation: 177/83

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration¹

This standard is issued under the fixed designation D 664; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval. This is also a standard of the Institute of Petroleum issued under the fixed designation IP177. The final number indicates the year of last revision.

This test method was adopted as a joint ASTM-IP standard in 1964.

This test method has been adopted for use by government agencies to replace Method 3106 of Federal Test Method Standard No. 791b. ASTM Test Method D 4739 has been developed as an alternative to the base number portion of D 664.

NOTE—An editorial correction was made in 12.3 December 21, 1989.

1. Scope

1.1 This test method covers procedures for the determination of acidic constituents in petroleum products and lubricants (Note 1). The test method resolves these constituents into groups having weak-acid and strong-acid, ionization properties, provided the dissociation constants of the more strongly acidic compounds are at least 1000 times that of the next weaker groups.

NOTE 1—In new and used oils, the constituents that may be considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, salts of ammonia and other weak bases, acid salts of polybasic acids and addition agents such as inhibitors and detergents.

1.2 The test method may be used to indicate relative changes that occur in an oil during use under oxidizing conditions regardless of the color or other properties of the resulting oil (Note 2). Although the titration is made under definite equilibrium conditions, the method is not intended to measure an absolute acidic property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid number is known.

NOTE 2—ASTM Test Method D 4739 was developed as an alternative to the base number portion of Test Method D 664 - 81. Base numbers obtained by this test method may or may not be numerically the same as those obtained by the base number portion of D 664 - 81.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved Dec. 21, 1989. Published January 1990. Originally published as D 664 - 42 T. Last previous edition D 664 - 87.

2. Referenced Documents

2.1 ASTM Standards:

- D974 Test Method for Acid and Base Number by Color-Indicator Titration²
- D1193 Specification for Reagent Water³
- D2896 Test Method for Total Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration⁴
- D3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration⁴
- D4739 Test Method for Base Number Determination by Potentiometric Titration⁵

3. Descriptions of Terms Specific to This Standard

3.1 *acid number*—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, required to titrate a sample in the solvent from its initial meter reading to a meter reading corresponding to a freshly prepared non-aqueous basic buffer solution or a well defined inflection point as specified in the test method.

3.2 *strong acid number*—the quantity of base, expressed as milligrams of potassium hydroxide per gram of sample, required to titrate a sample in the solvent from its initial meter reading to a meter reading corresponding to a freshly prepared non-aqueous acidic buffer solution or a well defined inflection point as specified in the test method.

4. Summary of Test Method

4.1 The sample is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide using a glass indicating electrode and a calomel reference electrode. The meter readings are plotted manually or automatically against the respective volumes of titrating solution and the end points are taken only at well defined

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 05.02.

⁵ Annual Book of ASTM Standards, Vol 05.03.

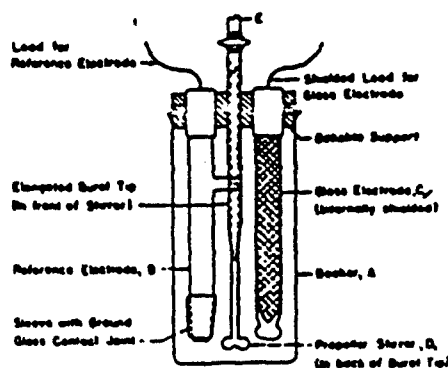


FIG. 1 Cell for Potentiometric Titration

inflections in the resulting curve. When no definite inflections are obtained, end points are taken at meter readings corresponding to those found for freshly prepared non-aqueous acidic and basic buffer solutions.

5. Significance and Use

5.1 New and used petroleum products may contain acidic constituents that are present as additives or as degradation products formed during service, such as oxidation products. The relative amount of these materials can be determined by titrating with bases. The acid number is a measure of this amount of acidic substance, in the oil-always under the conditions of the test. The acid number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation in service. Any condemning limits must be empirically established.

5.2 Since a variety of oxidation products contribute to the acid number and the organic acids vary widely in corrosion properties, the test cannot be used to predict corrosiveness of an oil under service conditions. No general correlation is known between acid number and the corrosive tendency of oils toward metals.

6. Apparatus

6.1 *Potentiometric Titrimeter*, automatic recording or manual.

6.2 *Glass Indicating Electrode*.

6.3 *Calomel Reference Electrode*, inverted sleeve type (Note 3).

6.4 *Stirrer, Buret, Stand, Titration Vessel*, as specified in Annex A2 are required. A typical cell assembly is shown in Fig. 1.

NOTE 3—Combination electrodes, because of their slow response, have been found unsuitable for this method.

7. Purity of Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chem-

ical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

7.3 *Buffer, Nonaqueous Acid*—Add 10 mL of buffer stock solution A to 100 mL of titration solvent. Use within 1 h.

7.4 *Buffer Nonaqueous Basic*—Add 10 mL of buffer stock solution B to 100 mL of titration solvent. Use within 1 h.

7.5 *Buffer Stock Solution A*—Accurately weigh 24.2 ± 0.1 g of 2,4,6-trimethyl pyridine (γ -collidine), and transfer to a 1-L volumetric flask containing 100 mL of isopropyl alcohol. Using a 1-L graduated cylinder, add to the flask, while continuously stirring its contents, $150/N_1 \pm 5$ mL of 0.2 *N* alcoholic HCl solution (N_1 being the exact normality of the HCl solution found by standardization). Dilute to the 1000-mL mark with isopropyl alcohol, and mix thoroughly. Use within 2 weeks.

7.6 *Buffer Stock Solution B*—Accurately weigh 27.8 ± 0.1 g of *m*-nitrophenol and transfer to a 1-L volumetric flask containing 100 mL of isopropyl alcohol. Using a 250-mL graduated cylinder, add to the flask while continuously stirring its contents, $50/N_2 \pm 1$ mL of 0.2 *N* alcoholic KOH solution (N_2 being the exact normality of the KOH solution found by standardization). Dilute to the 1000-mL mark with isopropyl alcohol and mix thoroughly. Use within 2 weeks.

7.7 *Hydrochloric Acid Solution, Standard Alcoholic (0.1 N)*—Mix 9 mL of hydrochloric (HCl, sp gr 1.19) acid with 1 L of anhydrous isopropyl alcohol. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration of approximately 8 mL (accurately measured) of the 0.1 *N* alcoholic KOH solution diluted with 125 mL of CO₂-free water.

7.8 *Hydrochloric Acid Solution, Standard Alcoholic (0.2 N)*—Prepare and standardize as directed in 7.7 but use 18 mL of HCl (sp gr 1.19).

7.9 *Isopropyl Alcohol, Anhydrous, (less than 0.1 % H₂O)* (Warning—see Note 4). If adequately dry reagent cannot be procured, it can be dried by distillation through a multiple plate column, discarding the first 5 % of material distilling overhead and using the 95 % remaining. Also drying may be accomplished using molecular sieves such as Linde type 4A, by passing the solvent upward through a molecular sieve column using 1 part of molecular sieve per ten parts of solvent.

NOTE 4: Warning—Flammable.

7.10 *2,4,6 Trimethyl pyridine (γ Collidine) (CH₃)₃C₅H₂N* (mol weight 121.18), conforming to the following requirements (Warning—see Note 5):

Boiling range	168 to 170°C
Refractive index n_D^{20}	1.4982 \pm 0.0005
Color	colorless

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopoeia."

NOTE 5—2,4,6-Trimethyl Pyridine (7 collidine): Warning—is hazardous if swallowed, breathed, or spilled on skin or eyes. Precaution: Wear chemical safety goggles, neoprene or rubber gloves and apron. Use only in a well-ventilated hood; or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.

7.10.2 Store the reagent over activated alumina and keep in a brown glass bottle.

7.11 *m*-Nitrophenol $\text{NO}_2\text{C}_6\text{H}_4\text{OH}$ (mol weight 139.11), conforming to the following requirements (Warning—see Note 6):

Melting point
Color

96 to 97°C
pale yellow

NOTE 6: Warning—*m*-Nitrophenol may be hazardous if swallowed, breathed, or spilled on skin or eyes. Wear chemical-safety goggles, neoprene or rubber gloves, and apron. Use only in a well-ventilated hood, or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.

7.11.1 Store the reagent in a brown glass bottle.

7.12 *Potassium Chloride Electrolyte*—Prepare a saturated solution potassium chloride (KCl) in water.

7.13 *Potassium Hydroxide Solution, Standard Alcoholic (0.1 N)*—Add 6 g of potassium hydroxide (KOH) to approximately 1 L of anhydrous isopropyl alcohol. Boil gently for 10 min to effect solution. Allow the solution to stand for 2 days and then filter the supernatant liquid through a fine sintered-glass funnel. Store the solution in a chemically resistant bottle. Dispense in a manner such that the solution is protected from atmospheric carbon dioxide (CO_2) by means of a guard tube containing soda lime or soda non-fibrous silicate absorbents and such that it does not come into contact with cork, rubber, or saponifiable stop-cock grease. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration of weighed quantities of potassium acid phthalate dissolved in CO_2 -free water.

7.14 *Potassium Hydroxide Solution, Standard Alcoholic (0.2 N)*—Prepare, store, and standardize as directed in 7.13, but use 12 to 13 g of KOH to approximately 1 L of isopropyl alcohol.

7.15 *Titration Solvent*—Add 500 mL of toluene (Warning—see Note 7) and 5 mL of water to 495 mL of anhydrous isopropyl alcohol. The titration solvent should be made up in large quantities, and its blank value determined daily by titration prior to use.

NOTE 7: Warning—Flammable.

8. Preparation of Electrode System

8.1 *Maintenance of Electrodes*—Clean the glass electrode (Note 8) at frequent intervals (not less than once every week during continual use) by immersing in cold chromic acid cleaning solution (Warning—see Note 9.) Drain the calomel electrode at least once each week and refill with fresh KCl electrolyte as far as the filling hole. Ascertain that crystallized KCl is present. Maintain the electrolyte level in the calomel electrode above that of the liquid in the titration beaker or vessel at all times. When not in use, immerse the lower halves of the electrodes in water. Do not allow them to remain immersed in titration solvent for any appreciable period of time between titrations. While the electrodes are not extremely fragile, handle them carefully at all times.

NOTE 8: Cleaning the electrodes⁷ thoroughly, keeping the ground-glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining repeatable potentials, since contamination may introduce uncertain erratic and unnoticeable liquid contact potentials.⁸ While this is of secondary importance when end points are chosen from inflection points in the titration curve, it may be quite serious when end points are chosen at arbitrarily-fixed cell potentials.

NOTE 9: Warning—Causes severe burns. A recognized carcinogen. Strong oxidizer. Contact with materials may cause fire. Hygroscopic.

8.2 *Preparation of Electrodes*—Before and after using, wipe the glass electrode thoroughly with a clean cloth, or a soft absorbent tissue, and rinse with water. Wipe the calomel reference electrode with a cloth or tissue, carefully remove the ground-glass sleeve and thoroughly wipe both ground surfaces. Replace the sleeve loosely and allow a few drops of electrolyte to drain through to flush the ground-glass joint (Note 8). Wet the ground surfaces thoroughly with electrolyte, set the sleeve firmly in place and rinse the electrode with water. Prior to each titration, soak the prepared electrodes in water for at least 5 min immediately before use, and touch the tips of the electrodes with a dry cloth or tissue to remove the excess of water.

8.3 *Testing of Electrodes*—Test the meter-electrode combination (Note 7) when first put into use, or when new electrodes are installed, and retest at intervals thereafter by dipping the electrodes into a well-stirred mixture of 100 mL of the titration solvent and 1.0 to 1.5 mL of 0.1 N alcoholic KOH solution. For the meter-electrode combination to be suitable for use, the potential between the electrodes should change by more than 480 mV from the potential between the same electrodes when dipped in the nonaqueous acidic buffer solution (Note 10).

NOTE 10: Considerably more sensitive electrodes are now available that will show a potential change of at least 590 mV under these conditions, and their use is recommended.

9. Standardization of Apparatus

9.1 *Determination of Meter Readings for the Nonaqueous Buffer Solutions Corresponding to Acid and Base End Points*—To ensure comparable selection of end points when definite inflection points are not obtained in the titration curve, determine daily, for each electrode pair, the meter readings obtained with freshly prepared nonaqueous acidic and basic buffer solutions.

NOTE 11: The response of different glass electrodes to hydrogen ion activity is not the same. Therefore, it is necessary to establish regularly for each electrode system the meter readings corresponding to the buffer solutions arbitrarily selected to represent acidic or basic end points.

9.2 Prepare the electrodes as described in 8.2, immerse them in the nonaqueous buffer solution, and stir for 5 min, maintaining the temperature of the buffer solution at a temperature within 2°C of that at which the titrations are to be made. Read the cell voltage. The readings so obtained are taken as the end points in titration curves having no inflection points.

⁷ For a detailed discussion of the need for care in preparation of the electrodes, see Lykken, L., Porter, P., Ruliffson, H. D., and Tuemmler, F. D., "Potentiometric Determination of Acidity in Highly Colored Oils," *Industrial and Engineering Chemistry, Analytical Edition*, IENAA, Vol 16, 1944, pp. 219-234.

10. Preparation of Sample of Used Oil

10.1 Strict observance of the sampling procedure is necessary since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

NOTE 12—As used oil may change appreciably in storage, test samples as soon as possible after removal from the lubricating system; and note the dates of sampling and testing.

10.2 Heat the sample (Note 13) of used oil to $60 \pm 5^\circ\text{C}$ in the original container and agitate until all of the sediment is homogeneously suspended in the oil. If the original container is a can or if it is glass and more than three-fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container.

NOTE 13—When samples are visibly free of sediment, the heating procedures described may be omitted.

10.3 After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

11. Procedure for Acid Number and Strong Acid Number

11.1 Into a 250-mL beaker or a suitable titration vessel, introduce a weighed quantity of sample as prescribed in Table 1 and add 125 mL of titration solvent (Note 14). Prepare the electrodes as directed in 8.2. Place the beaker or titration vessel on the titration stand and adjust its position so that the electrodes are about half immersed. Start the stirrer, and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution. If feasible adjust the meter so that it reads in the upper part of the millivolt scale, for example 700 mV.

NOTE 14—A titration solvent in which chloroform (Warning—May be fatal if swallowed. Harmful if inhaled. May produce toxic vapors if burned) is used in place of toluene and may be required to completely dissolve certain heavy residues of asphaltic materials.

11.2 Select the right buret, fill with the 0.1 *N* alcoholic KOH solution and place the buret in position on the titration assembly, take care that the tip is immersed about 25 mm in the liquid in titration vessel. Record the initial buret and meter (cell potential) readings.

11.3 Titration

11.3.1 *Manual Titration*—Add suitable small portions of 0.1 *N* alcoholic KOH solution and wait until potential equilibrium is reached (see Note 15). Record the buret and meter (cell potential) readings. At the start of the titration and in any subsequent regions (inflections) where 0.1 mL of 0.1 *N* KOH solution consistently produces a total change of

more than 30 mV (corresponding to 0.5 pH scale units) in the cell potential, add 0.05-mL portions. In the intermediate regions (plateaux) where 0.1 mL changes the cell potential less than 30 mV add larger portions sufficient to produce a total potential change approximately equal to, but not greater than 30 mV. Titrate in this manner until the potential changes less than 5 mV (corresponding to 0.1 pH scale units)/0.1 mL and the cell potential indicates that the solution is more basic than the freshly prepared nonaqueous basic buffer. Remove the titration solution, rinse the electrodes and buret tip with the titration solvent then with isopropyl alcohol and finally with grade reagent water. Immerse the electrodes in water for at least 5 min before using for another titration to restore the aqueous gel layer of the glass electrode. Store the electrodes in reagent water when not in use (see 8.1).

NOTE 15—Consider the cell potential constant when it changes less than 5 mV (corresponding to 0.1 pH scale units)/min. This may require approximately 1 to 2 min/100 mV change in potential (corresponding to 1.7 pH scale units) when adding 0.05-mL increments; 0.1-mL increments may require 3 to 5 min.

11.3.2 *Automatic Titration*—Adjust the apparatus in accordance with the manufacturer's instructions to comply with the potential equilibrium mode requirements established for the manual titration as explained in Note 10 and/or to provide a variable continuous delivery rate mode of titration of less than 0.2 mL/min during the titration and preferably 0.05 mL/min through the region of inflections and at the end point corresponding to that found for the freshly prepared standard nonaqueous basic buffer solution. Proceed with the automatic titration and record potentiometric curves or derivative curves as the case may be. Titrate in this manner with the 0.1 *N* alcoholic KOH solution until the potential become constant, for example, changing less than 5 mV/0.1 mL (automatic point) or when potential reading indicates that the solution is more basic than the freshly prepared nonaqueous basic buffer (pre-selected end point) solution.

11.3.3 On completion of the titration, rinse the electrodes and buret tip with the titration solvent, then with isopropyl alcohol and finally with reagent grade water. Keep the electrodes immersed in water for at least 5 min before reusing for another titration to restore the aqueous gel layer of the glass electrode. If electrodes are found dirty and contaminated proceed as in Section 8. Store the electrodes in reagent grade water when not in use.

11.4 *Blank*—For each set of samples, make a blank titration of 125 mL of the titration solvent. For manual titration, add 0.1 *N* alcoholic KOH solution in 0.05-mL increments, waiting between each addition until a constant cell potential is reached. Record the meter and buret readings when the former becomes constant after each increment. For automatic titration, proceed as in 11.3.2.

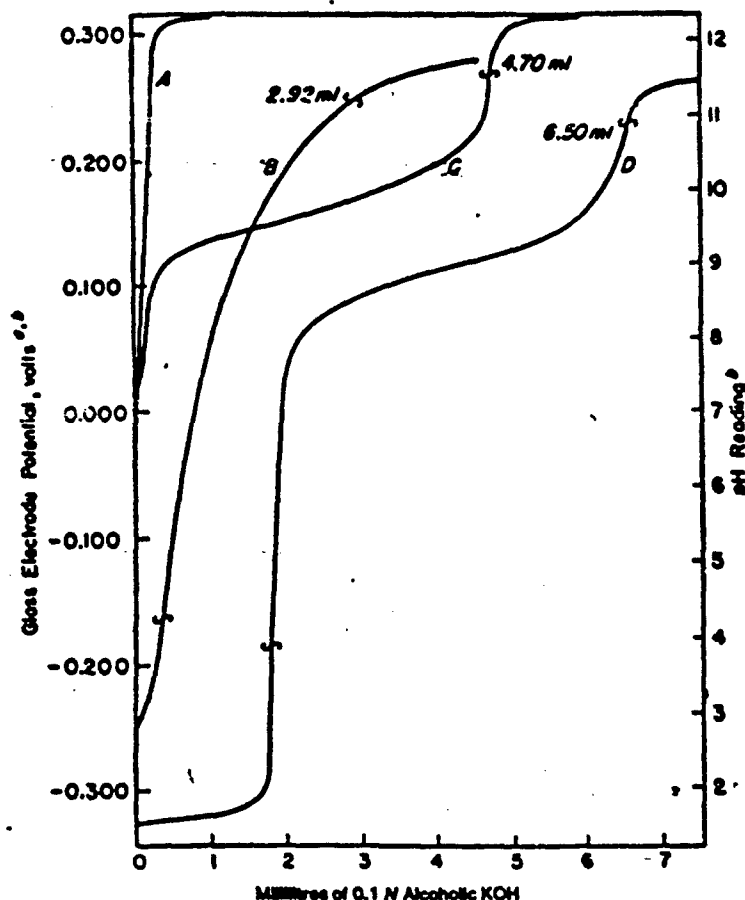
11.5 *Blank*—For each set of samples, make a blank titration of 125 mL of titration solvent, adding 0.1 *N* alcoholic HCl solution in 0.05-mL increments in a manner comparable to that specified in 11.4 for manual titration.

TABLE 1 Size of Sample

Acid Number	Size of Sample, g	Sensitivity of Weighing, g
0.05 to 0.9	20.0 ± 2.0	0.10
1.0 to 4.9	5.0 ± 0.5	0.02
5 to 19	1.0 ± 0.1	0.005
20 to 99	0.25 ± 0.02	0.001
100 to 250	0.1 ± 0.01	0.0005

12. Calculation

12.1 For manual titration, plot the volumes of the acid or base titrating solution, added against the corresponding



- A—Blank on 125 mL of titration solvent.
 B—10.00 g of used crankcase oil plus 125 mL of titration solvent. Since no sharp inflections are apparent the end points are chosen at the meter readings which were obtained with the two nonaqueous buffers.
 C—10.00 g of oil containing a weak acid plus 125 mL of titration solvent. The end point is chosen at the most vertical portion of the inflection.
 D—10.00 g of oil containing weak and strong acids plus 125 mL of titration solvent. The end points are chosen at the most vertical portions of the two inflections.

^a On some meters, the voltage sign is reversed.

^b In some instruments, the relation between glass electrode potential and pH reading is only approximately as shown.

FIG. 2 Illustrative Titration Curves

meter readings (see Fig 2). Mark as an end point, only well-defined inflection point (Note 16) being the closest to the cell voltages representing the freshly prepared nonaqueous acidic and basic buffers. If inflections are ill-defined or no inflection appears,^a mark the end points at those meter readings corresponding to the freshly prepared two nonaqueous buffers.

NOTE 16—One inflection point is generally recognizable by inspection whenever several successive 0.05-mL increments each produce a cell potential change greater than 15 mV (corresponding to 0.25 pH scale units) at least 30 % greater than those produced by previous or subsequent increments of the same size. Generally, definite inflection points may be discerned only in regions where increments of the same size are used.

12.2 For all acid titrations on used oils, mark as an end point, the point on the curve that corresponds to the freshly

prepared nonaqueous basic buffer end point (Note 17).

NOTE 17—The cooperative work done on acid number determinations on fresh oils, additive concentrates^a and used oils indicated well-defined inflection points for fresh oils and additive concentrates, and generally ill-defined inflections or no inflection points at all for used oils.

12.3 Calculate the acid number and strong acid number as follows:

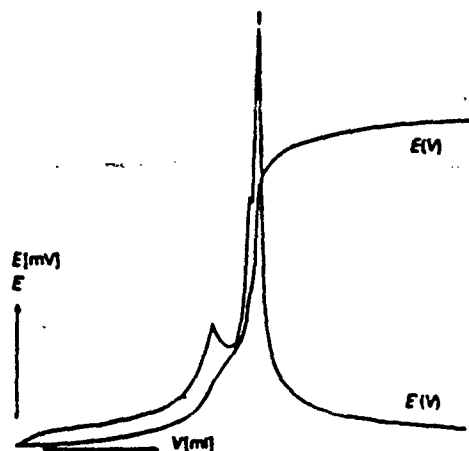
$$\text{Acid number, mg KOH/g} = (A - B) \times N \times 56.1/W$$

$$\text{Strong acid number, mg KOH/g} = (CN + Dm) \times 56.1/W$$

where:

A = alcoholic KOH solution used to titrate sample to end point that occurs at the meter reading of the inflection point closest to the meter reading corresponding to basic nonaqueous buffer, or in case of ill-defined or no inflection point, to meter reading corresponding to the basic nonaqueous buffer, mL,

^a Curve B in Fig. 2 is an example.



Note—Potential or pH curves are obtained by connecting a stepping motor recorder. This instrument records the titration curves—or their first derivatives—as a function of the delivered volume.

FIG. 3 Titration Curves and First Derivatives

- B = volume corresponding to A for blank titration, mL,
 N = normality of alcoholic KOH solution,
 n = normality of alcoholic HCl solution,
 W = sample, g,
 C = alcoholic KOH solution used to titrate the sample to end point that occurs at a meter reading corresponding to acid nonaqueous buffer, mL, and
 D = alcoholic HCl solution used to titrate solvent blank to end point corresponding to C , mL.

13. Report

13.1 Report the results as acid number or strong acid number as follows.

Acid number (D 664) = (result)

Strong acid number (D 664) = (result)

13.2 No modifications to this test method are permitted.

14. Precision and Bias

14.1 Acid Number:

14.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty:

Titration Mode	Fresh Oils and Additive Concentrates at Inflection Points		Used Oils at Buffer End Points	
	Manual	Automatic	Manual	Automatic
Percent of Mean	7	6	5	12

14.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty:

Titration Mode	Fresh Oils and Additive Concentrates at Inflection Points		Used Oils at Buffer End Points	
	Manual	Automatic	Manual	Automatic
Percent of Mean	20	28	39	44

14.2 Strong Acid Number:

14.2.1 Precision data have not been developed for strong acid number because of its rare occurrence in sample analysis.

14.3 *Bias*—The procedures in this test method have no bias because the acid values can be defined only in terms of the test method.

ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 The apparatus for manual titration shall consist of the following:

A1.1.1 *Meter*—A voltmeter or potentiometer that will operate with an accuracy of ± 0.005 V and a sensitivity of ± 0.002 V, over a range of at least ± 0.5 V, when the meter is used with the electrodes specified in A1.1.2 and A1.1.3, and when the resistance between the electrodes falls within the range from 0.2 to 20 M Ω . The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire operating range is produced by touching with a grounded⁹ lead, any part of the exposed

surface of the glass electrode, the glass electrode lead, the titration stand, or the meter. A desirable apparatus may consist of a continuous-reading electronic voltmeter with specified range, accuracy, and sensitivity, that is designed to operate on an input of less than 5×10^{-12} A when an electrode system having 1000 M Ω resistance is connected across the meter terminals, that is provided with a metal shield connected to the ground, and that is provided with a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interference from the presence of external electrostatic field.

A1.1.2 *Glass Electrode*—A pencil-type glass electrode (C, Fig. 1) 125 to 180 mm in length and 8 to 14 mm in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 to 3 mm. The end dipping into the solution shall be closed with a hemi-

⁹ "Grounded" or "connected to the ground" means connected through a resistance of not more than 100 Ω to a standard ground potential such as that of a water-service pipe.

sphere of Corning 715 glass or equivalent sealed on to the electrode tube, and the radius of this hemisphere shall be about 7 mm. The thickness of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 100 to 1000 M Ω at 25°C. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection with the inner surface of the hemisphere. The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of the electrical connection is greater than 50 000 M Ω .

A1.1.3 Calomel Electrode—A pencil-type calomel electrode (B, Fig. 1) 125 to 180 mm in length and 8 to 14 mm in diameter. This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 8 to 25 mm in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 2 to 20 mm beyond the sleeve. The ground surface shall be continuous and free of smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 1 mm in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated KCl electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than one drop in 10 min.¹⁰

¹⁰ Examples of suitable electrodes are: (a) Glass electrodes: Beckman 41263, Corning 476022, and Metrohm E 107. (b) Reference electrodes: Beckman 40463, Corning 476012, and Metrohm EA430.

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A1.1.4 Stirrer—A variable-speed mechanical stirrer of any suitable type, equipped with a glass, propeller-type stirring paddle (D, Fig. 1). A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. A magnetic stirrer is also satisfactory. If electrical stirring apparatus is used, it must be grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the course of titration.

A1.1.5 Buret—A 20-mL buret (E, Fig. 1) or appropriate size buret graduated in 0.05 mL division and calibrated with an accuracy of ± 0.02 mL. The buret shall have a glass stopcock and shall have a tip that extends 100 to 130 mm beyond the stopcock.

A1.1.6 Titration Beaker—A 250-mL beaker made of borosilicate glass (A, Fig. 1).

A1.1.7 Titration Stand—A suitable stand to support the electrodes, stirrer, and buret in the position shown in Fig. 1. An arrangement that allows the removal of the beaker or titration vessel without disturbing the electrodes, buret, and stirrer is desirable.

A1.2 Automatic titration system shall be generally in accordance with A2.1 and provide the following technical performance characteristics or features:

A1.2.1 Automatic adaption of the titration speed in the continuous titrant delivery mode to the slope of the titration curve with the capability of complying with the potential equilibrium specified and providing titration rates of less than 0.2 mL/min during titration and preferably 0.05 mL/min at inflections and at nonaqueous acid and basic end points.

A1.2.3 Interchangeable precision motor-driven burets with a volume dispensing accuracy of ± 0.01 mL.

A1.2.4 A record of the complete course of a titration by continuously printing out the relative potential versus volume of titrant added.

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